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INDEX TO VOLUME XXXVI.

INDEX OF NAMES.

ABDERHALDEN, E., *and* Meyer, C., Detection of Active Pepsin in the Intestines by Means of Elastin, 551

————————, *and* Schmidt, H., Use of Triketohydrindene Hydrate for the Detection of Proteins and their Derivatives, 514

Abelin, J., New Method for the Detection of Salvarsan, 452

Achalme, P., *and* Bresson, M., Method for Ascertaining whether a Liquid Contains One or More Enzymes, 110

————————————, Influence of the Viscosity on the Action of Invertase According to the Concentration of Saccharose, 416

Adams, A. B., Detection of the Substitution of Spirit for Aged Whisky, 546

———, L. H., *and* Johnston, J., Phenomenon of Occlusion in Precipitates of Barium Sulphate, and its Relation to the Exact Determination of Sulphates, 426

Adan, R., Researches on the Quantitative Analysis of Pentoses and Pentosans, and its Practical Applications, 293

Ageno, F., *and* Guicciardini, N., Estimation of Arsenic and Iron Salts in Mineral Waters, 466

Agulhon, H., Colorimetric Test for Alcohol in the Presence of Acetone, 592

Albrecht, A., *and* Müller, F., Estimation of Napthalene in Crude Coal-Gas, 510

Alexander, P., Caoutchouc Nitrosite and its Analytical Application, 287

Allen, I. C., Specifications and Sampling of Fuel Oil and Natural Gas, 555

Allesandri, P. E., Là Chimica delle Sostanze Alimentari, *Review*, 40

Amberger, C., Detection of Adulteration of Butter by Ewer's Method, 404

American Chemical Society, Analysis of Fats and Oils, 164

Andouard, —, *and* Bussard, L., Adulteration of Pepper Corns, 349

Andrews, A. E., The Determination of Codeine in Opium, 489

Andriska, V., *and* Liebermann, L. von, Method of Estimating the Quality of Wheaten Flour, 543

Anelli, G., Estimation of Sulphur by the Method of Carius, 296

Angerhausen, J., *and* Nottbohm, F. E., Wendler's Method for the Estimation of Fat in Milk, 14

Archbutt, L., Characteristics of Neutralised Olive-Residuum Oil, 116

Arnold, V., Colour Reaction of Proteins with Sodium Nitroprusside, 235

Arragon, C., New Constant in the Analysis of Pepper, 147

Ashley, H. E., *and* Emley, W. E., Errors in Determining Sizes of Mineral Grains and the Use of Surface Factors, 182

Atack, F. W., *and* Knecht, E., A New Method for the Volumetric Estimation of Molybdenum, 98

Atkinson, F. C., *and* McCandless, J. M., Bacteriological Method of Estimating Available Organic Nitrogen, 219

————, H. M., Modified Separatory Funnel and Washer for Heavy Liquids, 89

Auger, V., *and* Gabillon, M., New Method of Estimating Sulphuric Acid and Sulphates, 178

Autenrieth, W., *and* Funk, A., Colorimetric Estimation of Lactose in Urine and in Milk, 592
——, *and* Müller, G., Colorimetric Estimation of Sugar, Creatine, and Creatinine, in Urine, 591
Auzies, J. A. A., Analysis of a Substance Containing Carbon, Hydrogen, Oxygen, Nitrogen, and Sulphur, 553
——, Analysis of a Substance Containing Carbon, Hydrogen, Oxygen, Nitrogen, Sulphur, Chlorine, Bromine, and Iodine, 553
——, New Method of Elementary Analysis of Organic Substances Containing Carbon, Hydrogen, Oxygen, and Nitrogen, 553

Bach, W., Oil from Seeds of the Ash-Tree, 357
Bäcke, A. (*with* A. Wiborg *and* E. Röer), Analysis of Sweetened Condensed Milk, 138
Bacon, R. F., Detection and Estimation of Methyl and Ethyl Alcohols and Formic Acid, 507
——, Philippine Terpenes and Essential Oils, IV., 15
——, W., Note on the Gravimetric Estimation of Phosphorus in Milk, 583
Bailey, H. S., Electrically-Heated Vacuum Fractionation Apparatus, 253
Baker. J. L., *and* Day, F. E., The Chemical Behaviour and Preservative Action of Sulphites in Beer, 495
Bang, I., Estimation of Phosphoric Acid by Neumann's Method, 525
——, Preparation of Menthylglycuronic Acid, 505
——, Preparation of the Copper Solution used in Bang's Sugar Titration Method, 501
Barbier, P., Separation of Alumina from Ferric Oxide, 82
Bardach, B., An Inner Anhydride Reaction of Proteins, 514
——, Reaction of Aromatic Inner Anhydrides Depending on their Influence on the Crystalline Form of Iodoform, 458
——, *and* Silberstein, S., Polarimetric Estimation of Sugar by the Method of Jolles, 361
Barker, B. T. P., *and* Gimingham, C. T., Fungicidal Action of Bordeaux Mixtures, 351
Barrett, W. H., *and* Hartley, H., Determination of the Density of Liquids, 439
Barthel, C., Comparison of the Reductase Test with Other Hygienic Tests for Mi 346
Baskerville, C., *and* Hamor, W. A., Examination of Ether for Anæsthetic and Analytical Purposes, 407
——, *and* Stevenson, R., Analytical Examination of Commercial Oxygen, 469
——, Chemistry of Anæsthetics. III. Nitrous Oxide, 503
——, Oxidation of Ferrous Salts, 467
Batey, J. P., Notes on the Formation of Hypoiodites and their Action on Sodium Thiosulphate—A Source of Error in Certain Iodine Titrations, 132
Baubigny, H., Estimation of Very Small Quantities of Bromine in Presence of Chlorides and Iodides, 365
Baudisch, O., Quantitative Separation by Means of "Cupferron," 520
Baumann, P., Apparatus for Electrolytic Estimations with Mercury Cathode, 529
Baur, E., *and* Polenske, E., Method for the Separation of Starch and Glycogen, 501
Beadle, C., *and* Stevens, H. P., Rubber, *Review*, 443
——, Some Analyses of Hevea Latex, 6
Beck, K., *and* Lehmann, P., Estimation of Starch Syrup in Jams, etc., 280
Becker, R., Estimation of "Mineral Rubber" and Similar Products in Rubber Articles, 235
——, Notes on Hübener's Caoutchouc Tetrabromide, 235

Sewage Effluents Containing Nitrites, and of the Amount of Nitrite in Sewage Effluents and Water, 395
Burr, A., Composition of Dried Milk Powders, 279
Bussard, L., Adulteration of Linseed Cakes and Linseed Meals in Belgium, 218
——, Green Colour of Rapeseed Cakes, 411
——, *and* Andouard, —, Adulteration of Pepper-Corns, 349

Cahen, E., *and* Little, H. F. V., Waldemar Fischer's Modification of Volhard's Method for the Volumetric Estimation of Manganese, and its Comparison with Other Well-known Methods, 52
Cain, J. R., Determination of Vanadium in Vanadium and Chrome-Vanadium Steels, 475
——, Estimation of Manganese in Vanadium and Chrome-Vanadium Steels, 604
Cameron, F. K., The Soil Solution: The Nutrient Medium for Plant-Growth, *Review*, 574
Campbell, E. D., *and* Hurley, W. B., New Colorimeter, 478
Carles, P., Separate Estimation of Potassium Bitartrate and Calcium Tartrate, 234
Caro, N., (*and* Schück, B.), Analysis of Commercial Calcium Cyanamide, 76
Caron, H., *and* Raquet, D., Estimation of Nitrates by the Phenol Sulphonic Acid Method, 84
——, Examination of Bismuth Salicylate, 342
——, Estimation of Nitrates in Water by a Sulpho-Salicylic Reagent, 85
Carrez, D., Separation and Identification of Urobilin, 552
Casanova, C., Characteristic Colour Reaction of Ovolecithin, 460
Castell-Evans, J., Physico-Chemical Tables, Vol. II., *Review*, 192
Chamot, E. M., Pratt, D. S., *and* Redfield, H. W., Study of the Phenolsulphonic Acid Method for the Estimation of Nitrates in Water. III. Chief Sources of Error, 244
——, Study of the Phenolsulphonic Acid Method for the Estimation of Nitrates in Water. IV. A Modified Method, 244
Chance, E. M., *and* Blakeley, A. G., Accurate Technical Estimation of Calorific Value of Anthracite, 518
——, Estimation of Lead in Alloys Containing Antimony and Tin, 367
Chapin, R. M., Estimation of Nicotine in Nicotine Solutions and Tobacco Extracts, 544
——, Note on the Examination of Finnish Turpentine, 578
——, The Dimethyl Sulphate Test for Creosote Oil and Creosote Baths (Dips), 585
Chapman, A. C., A Further Contribution to the Question of Turpentine Substitutes, 211
——, The Detection of Traces of Hydrogen Cyanide, 269
——, H. G., *and* Welsh, D. A., Precipitin Reaction, 456
Charitschkoff, —, New Test for Ferrous Oxide, and the Separation of Iron from Aluminium, 373
Chéladzé, N., *and* Wunder, —, Separation of Aluminium from Beryllium, 426
Chercheffsky, N., Determination of Origin of Mineral Oils and their Products, 167
Chesneau, G., Analysis of Monazite Sands, 522
Chisholm, J., *and* Don, J., Modern Methods of Water Purification, *Review*, 261
Chouchak, D., *and* Pouget, I., Colorimetric Estimation of Phosphoric Acid, 525
Church, S. R., Methods for Testing Coal Tar and Refined Tars, Oils, and Pitches Derived therefrom, 298

Chwala, A., *and* Colle, H., Valuation of Higher Oxides of Lead, 366
Claassen, O., Estimation of Sulphurous Acid in Glucose Syrup, 543
Clark, S. G., *and* Ibbotson, F., Volumetric Estimation of Uranium, 248
Clarke, R. W., The Determination of the Amount of Dissolved Oxygen Absorbed by Sewage Effluents Containing Nitrites, and of the Amount of Nitrite in Sewage Effluents and Water, 393
Clausmann, P., Estimation of Bromine in Presence of Chlorides and Iodides, 241
Cockburn, J., *and* Black, J. W., The Estimation of Quinine as the Acid Citrate in Certain Organic Liquids, 396
Coffignier, C., Characteristics of Different Varieties of Dammar Resin, 422
Cohn, R., Lecithin, 510
———, Preservation of Fruit Juices with Hydrofluoric Acid, 218
Colle, H., *and* Chwala, A., Valuation of Higher Oxides of Lead, 366
Collingridge, W., Soda-Water, 16
Comey, A. M., Study of the Velocity of Detonation, 288
Cook, F. C., Comparison of Beef and Yeast Extracts of Known Origin, 64
———, Comparison of Meat and Yeast Extracts of Known Origin, 104
Cooper, E. A., *and* Morgan, G. T., Germicidal Action of Arsenic and Antimony Compounds on *Bacillus Typhosus*, 282
Coste, J. H., Note on Henry C. Frey's Method of Estimating Petroleum in Turpentine, 138
———, *and* James, B. R., Still Water Gas Calorimeter, 250
———, *and* Nash, L. M., A Further Contribution to the Question of Turpentine Substitutes, 207
———, *and* Powney, W. E. F., Testing of Incandescent Mantles, 125
Courmont, J., Sterilisation of Drinking Water by Means of Ultra-Violet Rays, 457
Coutts, F. J. H., Report to the Local Government Board on an Inquiry as to Condensed Milks, with Special Reference to their Use as Infants' Foods, 572
Craig, T. J. I., Estimation of Free Acid and Basic Alumina in Aluminium Salts, 236
Cranfield, H. T., The Effect of Feeding with Cocoanut Cake and Linseed Cake on the Composition of Butter-Fat, 445
Cribb, C. H., Observations on Some Methods of Estimating Cocoanut Oil and Butter in Butter and Margarine, 341
———, *and* Richards, P. A. E., Notes on the Analysis of Margarine, 327
Crispo, D., Loss of Soluble and Volatile Fatty Acids during the Ageing of Butter, 64
Curtman, L. J., *and* Rothberg, P., Application of the "Glow Reaction" to the Detection of the Platinum Metals, 434
———, Efficiency of Borax Bead Tests for Nickel and Cobalt in Admixture, 173

Dané, —, Detection of Nitrites in Water, 370
Daniel, K. C. R., *and* Nierstein, M., Use of Carbalkyloxy Derivatives for the Quantitative Estimation of Hydroxyl Groups, 227
David, —, Separation of Solid from Liquid Fatty Acids, 22
Davis, G. C., Dissolving Silicon Alloys, 178
Day, F. E., *and* Baker, J. L., The Chemical Behaviour and Preservative Action of Sulphites in Beer, 495
De Jong, A. W. K., Analysis of Coca Leaves, 274
Dede, L., Gravimetric Estimation of Nickel and Cobalt, 598
Degrazia, J. von, New Method for the Estimation of Nicotine in Tobacco, 280
———, Polarimetric Estimation of Nicotine in Tobacco Extracts, 279
Deiss, E., *and* Leysaht, H., Separation of Iron and Vanadium by Means of Ether, 522
Delehaye, H., Huiles Minérales (Pétroles, Benzols, Brais, Paraffines, Vaselines, Ozokérite), *Review*, 575

Delépine, S., Contribution to Study of Chemical Disinfectants, First and Second Papers, 69, 283
———, M., *and* Sornet, R., Separation and Estimation of Ammonia and Pyridine 508
Denigès, G., Detection of Ethyl Alcohol in the Presence of Methyl Alcohol, 18
——— Detection of Nitrates and Nitrites in Water, 432
——— New Reaction of Bromine, 427
——— New Reaction of Morphine, 66
——— New Test for Cupreïne, 113
Denis, W., Detection of Prune Juice and Caramel in Vanilla Flavouring Extracts, 281
Desch, C. H., The Chemistry and Testing of Cement, *Review*, 258
Desmoulière, E., Estimation of Citric Acid in Milk, 105
Dey, B. B., *and* Sen, H. K., Action of Hydrazine Sulphate on Nitrites, and a Method for the Estimation of Nitrite Nitrogen, 468
Dickert, M., "Perhydrol" for the Estimation of Total Sulphur in Coal-Gas, 309
Diefenthäler, O., *and* Müller, E., Simultaneous Volumetric Estimation of Iron and Vanadium, 475
Diethelm, B., *and* Müller, E., Estimation of Carbon and Sulphur in High-Grade Alloys of Tungsten, Molybdenum, and Vanadium, with Iron, 31
Dinslage, E., Analysis of Calcium Cyanamide and of Norwegian Nitre, 561
Dittrich, M., Determination of Ferrous Iron in Silicate Rocks, 602
——— Note on the Titration of Ferrous Oxide in Silicates by the Pebal-Dölter Method, 372
Dominicis, A. de, Estimation of the Age of Blood-Stains, 455
Dominikiewicz, M., Measuring Flask for Use in Determining Iodine Values of Oils, 379
Don, J., *and* Chisholm, J., Modern Methods of Water Purification, *Review*, 261
Donau, J., Gooch Crucible for Micro-Analysis, 180
Dorronsoro, B., Essential Oil of Spanish Marjoram (Thymus Mastichina, L.), 69
Downs, C. R., Distillation of Tars Containing Water, 171
Dox, A. W., Composition of Roquefort Cheese, 542
Dubois, W. L., *and* Lott, C. I., Estimation of Husk in Cocoa Powder, 275
Duclaux, J., Freezing Mixtures, 127
Dumas, W. C., Criticism of Magnesia Method for Estimation of Phosphoric Acid, 176
Dyer, B., Note on the Gravimetric Estimation of Phosphorus in Milk, 582

Ebler, E., Qualitative Analysis without the Use of Hydrogen Sulphide, 517
Eckert, A., *and* Meyer, H., The Fatty Oil and Wax Obtained from Coffee Berries, 106
Edgar, G., Precipitation of Vanadic Acid as Silver Vanadate, and Estimation of Phosphoric and Vanadic Acids in Presence of Each Other, 35
Edwards, M. G., *and* Orton, K. J. P., Detection and Estimation of Small Quantities of Acetic Anhydride in Acetic Acid, 417
Effront, J., Note on the Bulgarian Bacillus, 221
Egerton, A. C. G., Addition to the Buchner Funnel, 438
Egger, F., *and* Franzen, H., Estimation of Formic Acid, 355
Ehrlich, F., Preparation and Constitution of Caramel and its Estimation in Sugar Juices and Syrups, 273
Elliot, F. G., Volumetric Estimation of Sulphur in Iron and Steel, 374
Elvove, E., Estimation of Formaldehyde, 595
———, Use of Sulphur Dioxide in Checking Strengths of Volumetric Solutions of Iodine, Alkali, and Silver, 248
Emley, W. E., *and* Ashley, H. E., Errors in Determining Sizes of Mineral Grains and the Use of Surface Factors, 182

Enger, F., *and* Seiter, F. J., Identification of Cocaine and Substitutes, 405
Enz, H., Detection of Thujone in Absinthe, 495
Eriksson, E., Estimation of Glycyrrhizin and Sugars in Liquorice Powder and Extracts, 409
Esch, W., The Application of Bromination Methods for the Estimation of Caoutchouc in Vulcanised Products, 558
Essner, —, Analysis of Tobacco Juice and Extracts, and of Nicotine Salts, by the Method of Ulex, 545
Euler, H., The Chemistry of Enzymes (Allgemeine Chemie der Enzyme), *Review*, 534
Evans, J., *and* Scott-Smith, G. E., The Analytical and Microscopical Examination of Compound Liquorice Powder, 198
Ewins, A. J., Colour Reactions of Adrenaline and Allied Bases, 18

Fabris, U., Estimation of Water in Honey, 586
Fahrion, W., Estimation of Rosin in Fatty Oils, 557
————, Neats-Foot Oil, 512
Falciola, P., Separation of Liquid from Solid Fatty Acids—II., 114
————, Separation of Oleic Acid from Stearic and Palmitic Acids, 23
Favrel, G., Estimation of Iodine in Alcoholic Solutions, 235
Feder, E., Composition of Sauerkraut, 548
————, Detection of Invert Sugar in Honey, 586
Feiser, J. P., *and* Gooch, F. A., Estimation of Silver by Electrolysis of an Ammoniacal Solution of its Oxalate, 308
Feld, W., New Method of Estimating Tar and Tar Constituents in Gas, 299
————, Sodium Thiosulphate as Standard in Alkalimetry, 435
Fellenberg, T. Von, Colour Reactions of Aromatic Aldehydes and their Applicability in the Analysis of Spirits, 273
————, Detection of Resin in Glazed Coffee, 275
————, Higher Alcohols Occurring in Jamaica Rum, 411
Fendler, G., Analysis of Crude Caoutchouc, 159
Fenyvessy, B. von, Influence of Certain Vegetable Proteins on Wheat Gluten, 407
Fernandez, O., Spanish Oil of Turpentine, 589
Féry, C., Recording Thermo-Electric Calorimeter, 312
Fiechter, A., Reduction of Potassium Chloriplatinate, 526
Fincke, H., Chemical Differentiation between Fermentation Vinegars and "Vinegar Essence," Particularly with Respect to the Amount of Formic Acid, 455
————, Estimation of Formic Acid in Foods, 103, 496
————, Estimation of Pyridine in Vinegar, 414
Findlay, A., *and* Shen, B., Solubility of Carbon Dioxide in Beer, 404
Firth, J. B., *and* Myers, J. E., Apparatus for Precipitating, Filtering, and Drying in an Inert Gas, 313
Fischel, R., Histological-Chemical Detection of Peroxydase, 75
Fischer, F., Thiele, C., *and* Stecher, E., Rapid Electro-Analysis with Agitation by Means of a Current of Gas, 600
————, Rapid Electro-Analysis under Reduced Pressure, 601
Fisher, K., *and* Gruenert, O., Detection of Benzoic Acid in Flesh and Fats, 12
Fiske, A. H., *and* Jackson, C. L., Method for Purifying and Drying Organic Liquids by Wiping, 26
Flack, E. V., Citrate-Solubility of Phosphoric Oxide in Basic Slag, 32
Flanders, F. F., *and* Folin, O., Estimation of Benzoic Acid, 584
Fleurent, E., *and* Levi, L., Causes of Loss of Phosphoric Acid on Incineration in Presence of Organic Matter, 360

Fleurent, E., *and* Lévi, L., Estimation of Ash in Vegetable and Animal Substances, 237
Flohil, J. T., Estimation of Pentosans with the Aid of Fehling's Solution, 161
Florentin, D., *and* Marqueyrol, M., Estimation of Nitrogen in Nitrates, Gun-Cotton, etc., 243
Folin, O., *and* Flanders, F. F., Estimation of Benzoic Acid, 584
Foote, H. W., *and* Langley, R. W., Indirect Method for Determining Columbium and Tantalum, 239
————————————, Indirect Method for the Estimation of Tantalum and Columbium, 310
————————————, Separation of Tantalum and Columbium, 239
Forrest, C. N., Characters of Creosote and Tar Oils Available for Wood Preservation, 228
Fowler, G. J., Bacteriological and Enzyme Chemistry, *Review*, 260
Framm, F., Uniform Methods of Analysis for Portland Cement, 247
Francesconi, L., *and* Scarafia, —, Essential Oil of *Santolina Chamæcyparissus*, 589
Francis, C. K., Extraction Apparatus, 609
Frank, E., *and* Moeckel, K., Estimation of Sugar in Blood, 73
——— F., Proposed Uniform Method of Carrying out Comparative Tests for the Valuation of Raw Rubber, 423
Frankland, E. P., Estimation of Carbon and Nitrogen in Organic Compounds, 593
————— P. F., Bacteriology of Water: Its Present Position, 285
Franzen, H., Flask for Quantitative Fermentation Experiments, 568
—————, *and* Egger, F., Estimation of Formic Acid, 355
Fraps, G. S., Effect of Ignition on Solubility of Soil Phosphates, 371
Frazer, J. C. W., *and* Hoffman, E. J., Apparatus and Methods for Sampling and Analysis of Furnace Gases, 530
Fresenius, H., *and* Brinton, P. H. M. P., Estimation of Potassium as Potassium Platini-Chloride, 178
————, R., Application of "Cupferron" in Quantitative Analysis, 175
————, W., *and* Grünhut, L., Estimation of Lecithin in Oil, 166
Frey, W., *and* Wöhler, L., Estimation of Acidity in Hydrogen Peroxide Solutions, 82
Friedenthal, H., Quantitative Analysis of Mixtures by Differences in the Specific Gravities of their Components, 306
Fuhner, H., Detection of Colchicine in Toxicology, 73
Fuller, H. C., Estimation of Camphor, 593
Funk, A., *and* Autenrieth, W., Colorimetric Estimation of Lactose in Urine and in Milk, 592
Furnrohr, D., Estimation of Nitrogen in Liquids, 421
Fürth, O. von, Detection of Blood, 549

Gabillon, M., *and* Auger, V., New Method of Estimating Sulphuric Acid and Sulphates, 178
Gadais, J., *and* Gadais, L., Analysis of Liquorice Juice, 497
Gaebel, G. O., Detection of Salvarsan, 153
—————, Titration of "Salvarsan" with Iodine Solution, 350
Galletly, J. C., *and* Spence, D., Estimation of Caoutchouc as Tetrabromide, 509
——————, Spence, D., *and* Scott, J. H., Estimation of Rubber as Tetrabromide. Decomposition of the Bromide by Nitric Acid, 361
Gardner, J. A., *and* Buckmaster, G. A., Composition of the Gases of the Blood in Chloroform Anæsthesia, 154
Gatterbauer, J., Composition of the So-Called Gallisin (Glucosin) Occurring in Commercial Starch Syrup, 543
Gaubert, P., Differentiation of Minerals by Means of Colour Reactions, 242

Gautier, A., Use of Methyl Orange in the Colorimetric Estimation of Titanium, 310
Gawalowski, A., Apparatus for Flue Gas Analysis, 440
————, Microdistillation Apparatus, 37
Gaze, R., Estimation of Cantharidine in Tincture and Oil of Cantharides, 404
Gersten, E., *and* Ruff, O., Some Sources of Error in Gasometric Estimation of Nitrate by Schloesing's Method and of Nitrate by Piccini's Method, 525
Gilbard, J. F. H., A Reaction for Caulophyllin, 270
Gill, A. H., *and* Shippee, A. E., Estimation of Unsaponifiable Matter in Wool Oils, 168
Gimingham, C. T., *and* Barker, B .T. P., Fungicidal Action of Bordeaux Mixtures, 351
Gissing, G. E., The Spark Spectra of the Metals, *Review*, 41
Given, A., Bryan, A. H., *and* Straughn, M. N., Estimation of Sugars in Feeding-Stuffs, 453
Glaister, J., Arsenical Gas-Poisoning, *Review*, 321
Goldbaum, J. S., *and* Smith, E. F., Electrolytic Determination of Chlorine in Hydrochloric Acid with the Use of a Silver Anode and Mercury Cathode, 33
Golding, J., The "Neusal" Milk Test, 203
Goldsbrough, H. A., *and* Schidrowitz, P., Note on the Detection and Estimation of Small Quantities of Antimony, 101
Golubinzeff, A., Rapid Estimation of Ferric Oxide in Cement, 563
Gooch, F. A., *and* Boynton, C. N., Separation and Estimation of Barium in Presence of Calcium and Magnesium by the Action of Acetyl Chloride and Acetone on the Mixed Chlorides, 426
————, *and* Feiser, J. P., Estimation of Silver by Electrolysis of an Ammoniacal Solution of its Oxalate, 308
————, *and* Kuzirian, S. B., Application of Sodium Paratungstate in the Estimation of Carbon Dioxide in Carbonates and Nitrogen Pentoxide in Nitrates by Loss on Ignition, 526
Göpner, C., Dangers Connected with the Storage and Transport of Ferrosilicon, 302
Gottlieb, R., *and* Steppuhn, O., Quantitative Estimation of Morphine, 142
Goutal, E., *and* Mahler, P., Estimation of Carbon in Steel by Means of a Calorimetric Bomb, 562
Goy, —, *and* Stutzer, —, Estimation of Nitrate and Nitrite Nitrogen in Commercial Calcium Nitrate and Nitrite, 524
Grasser, G., Detection of Tannin in Solution, 412
————, Valuation of Logwood, 26
Greenish, H. G., The Microscopical Examination of Foods and Drugs, *Review*, 384
Greifenhagen, W., König, J., *and* Scholl, A., Estimation of Gelatin, 585
————————, Estimation of Starch, 588
Grenet, F., Testing the Porosity of Earthenware Filtering Bougies, 16
Griebel, L. C., Composition of the Pulp of Cassia Fistula, 214
Griffet, —, *and* Taurel, —, Estimation of Sublimed Sulphur in a Mixture of Different Kinds of Sulphur, 374
Griffiths, W., The Principal Starches Used as Food, *Review*, 488
Grimaldi, C., Reaction of Turpentine Oil, Wood Turpentine Oil, and Rosin Essence, 107
Grimbert, L., Separation of Urobilin from Urobilinogen, 223
Grimme, C., Characteristics of Conifer Fatty Oils, 511
————, Characteristics of Oils from Papilionaceous Plants, 231
————, Characteristics of Some Little-Known Vegetable Oils and Fats, 21
————, Composition of the Seeds of Leguminous Plants Grown in Togo and German East Africa, 345
————, Oil from the Seeds of *Xanthophyllum lanceolatum*, 406
Gross, A., *and* Sherman, H. C., Detection of Salicylic Acid, 463

Gruenert, O., *and* Fisher, K., Detection of Benzoic Acid in Flesh and Fats, 12
Grünhut, L., *and* Fresenius, W., Estimation of Lecithin in Oil, 166
Gudeman, E., Analyses of Evaporated Milk and Milk Products, 278
Guicciardini, N., *and* Adeno, F., Estimation of Arsenic and Iron Salts in Mineral Waters, 466

Hager, H., Composition of Goat's Milk, and the Detection of Goat's Milk in Cow's Milk, 218
Hailer, E., Experiments on the Antiseptic Properties of Sulphurous Acid, Sulphites, and Complex Compounds of Sulphurous Acid, 285
Hall, C., *and* Munroe, C. E., A Primer on Explosives for Coal-Miners, *Review*, 380
Hamill, J. M., I., On the Bleaching of Flour and the Addition of So-Called "Improvers" to Flour, 254
———, II., On the Presence of Calcium Sulphate in Baking Powder and Self-Raising Flour, 255
———, Report to the Local Government Board on the Nutritive Value of Bread Made from Different Varieties of Flour, 479
Hamilton, H. C., Germicidal and Insecticidal Values of Different Soaps, with or without Associated Active Agents, 505
Hamor, W. A., *and* Baskerville, C., Examination of Ether for Anæsthetic and Analytical Purposes, 407
Hankin, E. H., Tests for Cocaine and Certain Other Anæsthetics, 2
Hanǔs, J., *and* Soukup, A., Estimation of Copper by Means of Hypophosphorus Acid, 302
———, *and* Thian, J., Estimation of Cocoanut Oil in Lard by Means of the "Ethyl Ester Value," 106
Harrison, W., Starch-Iodine Reaction, 28
Hartel, F., *and* Solling, J., Analysis and Composition of Jams, 144
Hartley, H., *and* Barrett, W. H., Determination of the Density of Liquids, 439
Hartmann, M. L., *and* Benner, R. C., Rapid Determinations and Separations by Means of the Mercury Cathode and Stationary Anode, 83
Harvey, H. W., Note on the Composition of Soot, 120
Hasenbäumer, J., König, J., *and* Meyering, H., Application of the Osmotic Pressure and Electrical Conductivity in the Examination of Soils, 184
———, Determination of the Electrical Conductivity of Soils, 183
Hattensaur, G., Estimation of Arsenic in Pyrites, 362
Hawk, P. B., Howe, P. E., *and* Rutherford, T. A., Preservation of Fæces, 74
Hawkins, E. M., Note on Gorgonzola Cheese, 63
Hayworth, W. P., *and* Pickles, S. S., Composition of Para Rubber-Seed Oil, 491
———, Composition of the Fat from the Seeds of *Lophira alata* (Banks), 493
Heath, G. L., Estimation of Arsenic and Antimony in Copper, 171
———, Exact Electrolytic Assay of Refined Copper. I. Standard Method, 172
Heczko, A., Estimation of Tartaric Acid, 148
———, Estimation of Total Tartaric Acid, 148
Heide, R. von der, Condenser and Extraction Apparatus, 378
Heiduschka, A., *and* Kaufmann, G., Volatile Acids of Honey, 277
———, *and* Reuss, A., Estimation of Arsenic in Schweinfurt Green, 363
Hempel, H., Beythien, A., Simmich, P., Schwert, W., *and* Wiesemann, C., Estimation of Formaldehyde by the Ammonia Method, 421
Henblein, O., *and* Tillmanns, J., Titration of Alkali Carbonates in Presence of Alkali Hydroxides or Bicarbonates, 362

Henriet, H., *and* Bouyssy, M., Method of Measuring the Degree of Vitiation of the Atmosphere, 363

Hepner, A., Detection of Cocoanut Oil in Butter by Means of Fendler's Method, 451

Hercod, E., *and* Maben, T., Comparative Studies on the Valuation of Pepsin, 155

Herold, J., Analysis of Gelatine, 289

———, Junr., Analysis of Gelatins, 116

Herrmann, F., Estimation of Formaldehyde, 114

Herting, O., Simple Volumetric Method for Testing Spiritus Ætherus Nitrosi, 454

Hertkorn, J., Toxic Substances in Vegetable Fats and Butters, 65

Herzog, E., Detection of Fusel Oil in Brandy, 216

Hetper, J., Action of Permanganate on Organic Substances, 422

———, Influence of Potassium Permanganate upon Organic Bodies, 232

Heyl, F. W., *and* Raiford, L. C., Analysis of Zygadenus Intermedius ("Death Camas"), 156

Hibbert, E., *and* Knecht, E., A Volumetric Method for the Estimation of Tungsten, 96

———, The Influence of Pertitanic Acid on the Estimation of Iron in Minerals, 304

———, Volumetric Estimation of Quinones by Means of Titanium Trichloride, 80

Hicks, E. F., Colour Reactions of Certain Resins with Halphen's Reagent for Colophony, 170

———, W. B., Use of Sulphur Monochloride in the Determination and Analysis of Rare Earth Minerals, 567

Hill, C. A., The Analytical and Microscopic Examination of Compound Liquorice Powder, 198

Hillebrand, W. F., *and* Blum, W., Estimation of Manganese by the Bismuth Method, 430

Hinds, J. I. D., Qualitative Chemical Analysis from the Standpoint of Solubilities, Ionisation, and Mass Action, *Review*, 259

———, Sulphite Method for Separating and Identifying Calcium and Strontium, 301

Hincks, E., Note on Gorgonzola Cheese, 61

———, Observations on Some Methods of Estimating Cocoanut Oil and Butter in Butter and Margarine, 333

———, The Determination of the Amount of Dissolved Oxygen Absorbed by Sewage Effluents Containing Nitrites, and of the Amount of Nitrite in Sewage Effluents and Water, 395

Hinrichsen, W., *and* Kindscher, E., Direct Estimation of Caoutchouc as Tetrabromide, 224

Hirschberg, M., Detection of Amylolytic Enzymes in Fæces, 74

Hodgson, T. R., *and* Russell, E., Note on the Composition of British Wines, 60

Hoffman, E. J., *and* Frazer, J. C. W., Apparatus and Methods for Sampling and Analysis of Furnace Gases, 530

Hoft, H., Detection of the Use of Alkali Carbonates in Cheese Manufacture, 218

Holde, D., *and* Marcusson, J., Estimation of Free Fatty Acids in the Presence of Soaps of the Alkalis and Alkaline Earths, 595

———, *and* Meister, F., Estimation of Rosin in the Residues from the Distillation of Coal Tar, 463

———, *and* Meyerheim, G., Estimation of Tarry Substances in Dark Mineral Cylinder Oils, 298

Holland, E. B., Estimation of Arsenic in Insecticides, 237

Holmes, J. A., Sampling of Coal in the Mine, 527

Houston, A. C., Vitality of Typhoid Bacilli in River Water, with Special Reference to the Question of Storage, 185
Howard, C. D., Estimation of Essential Oils, 288
Howe, P. E., Rutherford, T. A., *and* Hawk, P. B., Preservation of Fæces, 74
Hübener, G., Direct Estimation of Caoutchouc in Vulcanised Rubber Articles, 75, 113
Huber, H. von, *and* Marcusson, J., Detection of Train Oil in Oils, Fats, and Soaps, 359
Hulett, G. A., *and* Perdue, W. H., Exact Method for the Electrolytic Estimation of Metals, 370
Hurley, W. B., *and* Campbell, E. D., New Colorimeter, 478
Huss, H., Quantity of Husk and Germ in Cocoa Products, 214
Hutchin, H. W., The Assay of Wolfram Concentrate, 398
Hüttner, C., *and* Mylius, F., Use of Ether in the Analysis of Metals, 431
Hyde, A. W. T., *and* Price, T. S., Adaptation of the Tap-Funnel to Rapid Electro-Analysis with Stationary Electrodes, 313

Ibbotson, F., *and* Clark, S. G., Volumetric Estimation of Uranium, 248
Ingle, H., Notes on Linseed and Other Oils, 278
Ishida, M., *and* Tollens, B., Estimation of Pentosans and Methylpentosans in Cereals and Wood-Fungi, 460
Iwanow, W. N., Estimation of Copper in Pyrites and in Burned Pyrites, 366

Jaboin, A., International Standard Units for the Measurement of the Radio-Activity of Radium, 35
Jackson, C. G., Some Forms of Glass Manometer, 437
———, C. L., *and* Fiske, A. H., Method for Purifying and Drying Organic Liquids by Wiping, 26
Jámbor, J., Elimination of Ammonium Salts Prior to Estimation of Alkali Metals, 30
James, B. R., *and* Coste, J. H., Still Water Gas Calorimeter, 250
———, C., *and* Pratt, L. A., New Method for Separation of Cerium, 518
Jamieson, G. S., Volumetric Method for Antimony in Alloys, 299
Javillier, M., Estimation of Atropine. Titration of the Alkaloids in Belladonna Extracts, 142
Jensen, H. R., Analytical Examination of Linseed Oil, 407
Johnston, J., *and* Adams, L. H., Phenomenon of Occlusion in Precipitates of Barium Sulphate, and its Relation to the Exact Determination of Sulphate, 426
Jolles, A., New Method of Estimating Sucrose in the Presence of Other Sugars, 28
Jong, A. W. K. de, Analysis of Coca Leaves, 274
Jörgensen, A., Micro-Organisms and Fermentation, *Review*, 191
———, G., Estimation of Phosphoric Acid as Magnesium-Ammonium Phosphate, 434
Jorissen, A., Identification of Veronal, 501
———, Reaction of Sparteine, 559
Jungfleisch, E., Method of Destroying Organic Matters in Testing for Mineral Poisons, 110

Kakiuchi, S., Estimation of Fat in Pathological Urine, 417
Kallauner, O., Estimation of Magnesium as Oxide, 604
Kappen, H., Analysis of Calcium Cyanamide, 560
Kast, H., Sensitiveness Tests by the Impact Machine (Drop Hammer), 314
Kastle, J. H., Acids Suitable for Use as Standards in Acidimetry, 25
Katz, J., Volumetric Estimation of Quinine in Drugs, etc., 143
Kaufmann, G., *and* Heiduschka, A., Volatile Acids of Honey, 277

Kaysser, A., Analysis of Bog-Iron Ores, 123

Keimatsu, S., Composition of Soya-Bean Oil, 513

Kempf, R., Automatic Extraction of Aqueous Liquids by Means of Specifically Lighter Solvents, 87

Kereszty. G. von, *and* Tangl, F., Wet Method for the Estimation of Carbon in Organic Substances, 354

Kesara-Menon, A., Some Indian Oils and Fats, 108

Kessler, J. J., *and* Mathiason, G. K., Interpolation Method of Oil Analysis, 166

Kindscher, E., *and* Hinrichsen, W., Direct Estimation of Caoutchouc as Tetrabromide, 224

Klapproth, W., Analysis of Lactic Acid, 556

Kleist, G., Analysis of Aluminium and Aluminium Alloys, 425

Kling, A., Racemic Acid as a Reagent (Precipitant of Calcium and Strontium), 365

Knecht, E., *and* Atack, F. W., A New Method for the Volumetric Estimation of Molybdenum, 98

———, *and* Hibbert, E., A Volumetric Method for the Estimation of Tungsten, 96

——————, The Influence of Pertitanic Acid on the Estimation of Iron in Minerals, 304

——————, Volumetric Estimation of Quinones by Means of Titanium Trichloride, 80

Kneip, A., Ney, N., *and* Reimers, F., Quantitative Estimation of Cantharidine in Cantharides and in Tinctures, 343

Knorr, L., *and* Schubert, H., Colorimetric Method for the Quantitative Estimation of Enols in Allelotropic Mixtures, 554

Kober, P. A., Differentiation between Amino-Acids and Peptones or Peptides, 593

Koenig, P., Colorimetric Estimation of Minute Quantities of Chromium, 238

Kohn-Abrest, E., Some Uses of Amalgamated Aluminium in Chemical Analysis, 516

———, W., Estimation of Nicotine in Tobacco Extracts, 348

König, J., Hasenbäumer, J., *and* Meyering, H., Application of the Osmotic Pressure and Electrical Conductivity in the Examination of Soils, 184

——————————, Determination of the Electrical Conductivity of Soils, 183

———, Scholl, A., *and* Greifenhagen, W., Estimation of Gelatin, 585

Estimation of Starch, 588

Kooper, W. D., Oxydase and Reductase Occurring in Cow's Milk, 16

Koppenhague, R., Reagent Paper for the Volumetric Estimation of Zinc, 126

Köpke, O., *and* Polenske, E., Estimation of Potassium Nitrate in Meat, 217

Korneck, O., Critical Investigation of the Analytical Methods for the Estimation of Caoutchouc, 158

———, Critical Investigations on the Analytical Methods for Raw Rubber, 27

Köster, J., Electrolytic Estimation of Manganese, 177

Kraemer, H., A Textbook of Botany and Pharmacognosy, *Review*, 130

Kreidl, A., *and* Lenk, E., Determination of the Specific Gravity of Very Small Quantities of Milk, 500

Kreis, H., Estimation of the Reichert-Meissl Value of Fats by Means of Glycerol-Potassium Hydroxide, 542

Kreuzer, C., Estimation of Starch in Brewers' Grains, 500

Krog, K., *and* Sebelien, J., Quantitative Estimation of Nitric Acid in Vegetable Products, 163

Kroll, —, *and* Rupp, E., Analysis of Calcium Hypophosphite, 605

Kronberger, H, Opsonin Reaction, 455

Krulla, R., Direct Measurement of Changes of Vapour Pressure, and Detection of Definite Compounds in Mixed Liquids by its Means, 379
Kurtenacker, A., Elementary Analysis, 516
Kustenmacher, M., Chemistry of Honey Formation, 223
Kutscher, F., Soluble Basic Constituents of the Mushroom, 347
Kuzirian, S. B., *and* Gooch, F. A., Application of Sodium Paratungstate in the Estimation of Carbon Dioxide in Carbonates and Nitrogen Pentoxide in Nitrates by Loss on Ignition, 526

Lafar, F., Technical Mycology, *Review*, 92
Laloue, G., Essential Oil of Orange Flowers, 67
——, —— of Schinus Molle (L.), 68
Lander, G. D., *and* Walden, A. E., The Detection of Traces of Hydrogen Cyanide, 266
Landrum, R. D., Analysis of Enamel, 33
Langley, R. W., *and* Foote, H. W., Indirect Method for Determining Columbium and Tantalum, 239, 310
——, Separation of Tantalum and Columbium, 239
Langmuir, A. C., *and* White, F. S., Analysis of Shellac and Shellac Varnish, 464
Lathrop, E., *and* Schreiner, O., Dihydroxystearic Acid in Good and Poor Soils, 504
Law, D. J., *and* Sand, H. J., Electrometric Method for the Estimation of the Acidity of Tan Liquors, 121, (Part II.) 515
LaWall, C. H., *and* Leffmann, H., Sulphur Dioxide in Commercial Gelatins, 271
Laxa, O., Estimation of Catalase in Milk, 284
Lechner, G., Estimation of Ozone by Means of Alkaline Potassium Iodide Solution, 525
Lecocq, E., Carbonylferrocyanides: Their Extraction and Analysis, 157
——, Estimation of Cyanogen Compounds in the Gases from Coke Furnaces, 77
Leffmann, H., *and* LaWall, C. H., Sulphur Dioxide in Commercial Gelatins, 271
Lehmann, F., *and* Rupp, E., Decomposition of Silicates by Fusion with Boric Acid as Applied to Qualitative Analysis, 371
——, New Method of Estimating Nitrites, 307
——, Simplified Method for the Estimation of Arsenic in Atoxyl and Arsacetin, 273
——, P., *and* Beck, K., Estimation of Starch Syrup in Jams, etc., 280
Leiper, R. T., *and* MacFadden, A. W. J., On a Parasite Condition (Onchocerciasis) Met with in Australian Beef, 129
Lenk, E., *and* Kreidl, A., Determination of the Specific Gravity of Very Small Quantities of Milk, 500
Lenz, W., Adulterant of African Sandal Wood Oil, 146
——, Testing of Camphor, 343
——, *and* Richter, E., Detection of Perborates and Similar Compounds, 470
——, *and* Schoorl, N., Micro-Chemical Test for Sodium, 372
Leroide, J., *and* Tassilly, E., Relative Proportions of Arsenic in Marine Algæ and Products Derived from Them, 218
Lessing, R., The Detection of Traces of Hydrogen Cyanide, 269
Leuchter, M., Modified Cobalt Reaction for the Detection of Very Small Quantities of Hydrogen Peroxide, 598
Levi, C., Detection of Animal Sizing in Paper, 290
——, L., *and* Fleurent, E., Causes of Loss of Phosphoric Acid on Incineration in Presence of Organic Matter, 360
——, Estimation of Ash in Vegetable and Animal Substances, 237
Leysaht, H., *and* Deiss, E., Separation of Iron and Vanadium by Means of Ether, 522
Lidoff, A. P., Characteristics of Certain Oils, 21

Leibermann, L. von, Apparatus for the Estimation of the Melting-Point of Fats, 569
——————, Laboratory Apparatus for the Production of a Constant Current of Ozonised Air, 479
——————, *and* Andriska, V., Method of Estimating the Quality of Wheaten Flour, 543
Liebert, F., Simple Laboratory Suction Pump, 532
Liesegang, R. E., Incineration of Microtome Sections, 74
Little, H. F. V., *and* Cahen, E., Waldemar Fischer's Modification of Volhard's Method for the Volumetric Estimation of Manganese, and its Comparison with Other Well-known Methods, 52
Lloyd, C. F., Bell, W. J., *and* Robinson, R. A., The Sale of Food and Drugs Acts, *Review*, 325
Loebell, H., Estimation of the Acidity of Fats and Oils, particularly Lubricating Oils, 290
——————, Proportion of Asphalt Insoluble in Alcohol-Ether in Mineral Lubricating Oils, 418
Loele, W., Colorimetric Detection of Oxidising Substances in Animal Organs, 152
Loevy, J., Pot Assay of Gold and Silver in Ores Containing much Copper, 240
Lohmann, D., Asphaltum from Mexican Petroleum, 353
——————, Technical Analysis of Petroleum Asphalt, 294
Lohrisch, H., Estimation of Cellulose by Simon and Lohrisch's Method, 77
Loomis, H. M., Report on the Solubility, Extraction, and Reactions of Certain Colouring Matters Used in Foods, 186
Loram, H. Y., Simple Method of Decomposing Refractory Tin Ores, 309
Lott, C. I., *and* Dubois, W. L., Estimation of Husk in Cocoa Powder, 275
Louïse, E., Analysis of Cod-Liver Oil by Means of its Miscibility Curve, 275
——————, Use of Miscibility Curves in the Examination of Essential Oils and Balsams, 556
Low, W. H., The Precipitation of Nickel Compounds and Preparation of Spongy Nickel, 539
Lucas, A., Report on the Work of the Laboratories, Survey Department, Egypt (1910), 532
Lührig, H., *and* Scholz, A., Examination of Honey by Fiehe's Reaction, 451
Lund, R., Analysis of Honey, with Special Reference to the Nitrogenous Constituents, 277

Maben, T., *and* Hercod, E., Comparative Studies on the Valuation of Pepsin, 155
Macara, T., Observations on Some Methods of Estimating Cocoanut Oil and Butter in Butter and Margarine, 341
MacFadden, A. W. J., *and* Leiper, R. T., On a Parasite Condition (Onchocerciasis) Met with in Australian Beef, 129
Machiedo, L., *and* Moser, L., Separation of Strontium from Calcium, 247
Maclaurin, J. S., Annual Report of the Dominion Laboratory, Wellington, New Zealand (1909), 91
Madelung, W., Estimation of the Activity of Oxidising Agents by Means of the Quinonoid Imonium Salts of Benzidine, 233
Magnin, G., Method of Destroying Organic Matter by Means of Bromine, particularly applicable to Toxicological Analysis, 592
Mahler, P., *and* Goutal, E., Estimation of Carbon in Steel by Means of a Calorimetric Bomb, 562
Main, H., Estimation of Ash in Sugar and Syrups by the Determination of Electrical Conductivity, 298
Malenfant, R., Analysis of Gum Arabic Pastilles, 344
Mallat, A., Characteristic Constituent of Natural Vichy Salts, 85

Malvezin, P., Estimation of Tannin in Wines, 412
Mameli, E., Influence of Certain Compounds upon the Colour Reactions of Strychnine, 296
Mann, E. A., Analysis of Potable Spirits, and its Bearing on the Definition of Whisky (Report), 441
————, Composition of the Gases Caused by Blasting in Mines (Report), 321
Marcusson, J., Composition of Mineral Lubricating Oils of High Boiling-Point, 461
————, Detection of Petroleum Pitch in Fat Pitch, 462
————, Examination of Asphaltum, 225
————, Examination of Wool-Grease Oleines, 29
————, *and* Holde, D., Estimation of Free Fatty Acids in the Presence of Soaps of the Alkalis and Alkaline Earths, 595
————, *and* Meyerhelm, G., Detection of Graphite in Lubricants, 356
————, *and* Von Huber, H., Detection of Train Oil in Oils, Fats, and Soaps, 359
Marqueyrol, M., *and* Florentin, D., Estimation of Nitrogen in Nitrates, Gun-Cotton, etc., 243
————, Estimation of Camphor in Smokeless Powder, 420
————, Estimation of Chlorides, Chlorates, and Perchlorates in the Presence of Each Other, 365
Marrs, L. E., *and* Metzger, F. J., Rapid and Accurate Method for Estimation of Manganese, 368
Martindale, W. H., Organic Analysis Chart, *Review*, 40
————, *and* Westcott, W. W., The Extra Pharmacopœia, *Review*, 38
Maryott, C. H., Use of Metallic Potassium in the Estimation of Halogens in Benzene Derivatives, 287
Masson, I., Use of Calcium Carbide for Determining Moisture, 112
Mathewson, W. E., *and* Seeker, A. F., Estimation of Iodine in Organic Compounds, and its Separation from Other Halogens, 118
Mathiason, G. K., *and* Kessler, J. J., Interpolation Method of Oil Analysis, 166
Maudsley, F., Ross, R., *and* Race, J., Examination of Shrewsbury and Knapp's Process for the Estimation of Cocoanut Oil, 195
Mayer, F., Analysis of Graphite, 562
M'Cabe, C. R., Colorimetric Method for Vanadium in Iron and Steel, 473
McCandless, J. M., *and* Atkinson, F. C., Bacteriological Method of Estimating Available Organic Nitrogen, 219
McCaughey, R. S., Quantitative Estimation of Volatile Fatty Acids in Fæces, 505
McCrae, J., Kobert's Reagent as a Test for Salicylic Acid, 540
McDermott, F. A., Hippuric Acid as a Cause of the Failure of the Spectroscopic Test for Hæmoglobin in Urine, 417
Mdivani, B., Estimation of Tungsten, 126
————, Estimation of Tungsten by Means of Stannous Chloride, 310
Mecklenburg, W., *and* Winterfeld, G., Detection of Mineral Oil in Wool-Grease Oleine, 29
Meister, F., *and* Holde, —, Estimation of Rosin in the Residues from the Distillation of Coal-Tar, 463
————, —, Iodine Values of Linoxyn, 24
Mellett, R., Accurate Estimation of Nicotine in Tobacco and in Green Plants of *Nicotiana Tabacum*, 348
Menge, G. A., Melting-Point Determinations, 438
Menzies, A. W. C., *and* Smith, A., Static Method for Determining Vapour Pressures of Solids and Liquids, 36
Merriman, R. W., *and* Wade, J., Apparatus for the Maintenance of Constant Pressures Above and Below the Atmospheric Pressure, Application to Fractional Distillation, 441

Mestre, P. C., Sulphurous Acid in White Wines, 351
Metzger, F. J., *and* Marrs, L. E., Rapid and Accurate Method for Estimation of Manganese, 368
Meyer, C., *and* Abderhalden, E., Detection of Active Pepsin in the Intestines by Means of Elastin, 551
———, H., *and* Eckert, A., The Fatty Oil and Wax Obtained from Coffee Berries, 106
———, J., Seychelles Cinnamon Bark, 217
———, R. J., Detection and Estimation of Thorium by Means of Iodic Acid (Separation of Thorium from Scandium), 436
Meyerheim, G., *and* Holde, D., Estimation of Tarry Substances in Dark Mineral Cylinder Oils, 298
————————, *and* Marcusson, J., Detection of Graphite in Lubricants, 356
Meyering, H., König, J., *and* Hasenbäumer, J., Application of the Osmotic Pressure and Electrical Conductivity in the Examination of Soils, 184
————————————————————, Determination of the Electrical Conductivity of Soils, 183
Michel, F., Detection of Blood by Means of Leuco-Malachite Green, 284
—————, Hæmochromogen and the Spectroscopic Differentiation between Oxyhæmoglobin and Carbon Monoxide Hæmoglobin, 550
—————, Influence of Metals in Presence of Hydrogen Peroxide on Blood Reagents, 352
—————, Modification of Heller's Ring Test for the Detection of Albumin in Urine, etc., 152
Micko, K., Analysis of Meat-Juices, 13
—————, Coagulating-Points of Egg, Serum, and Milk Albumins, and of Serum Globulin, in Ammonium Sulphate Solutions, 416
Mielck, H., Effect of the Glycerol Method of Saponification upon the Fatty Acids, 425
Milbauer, J., Device for Accurately Reading a Burette, 312
——————, *and* Quadrat, O., Ferric Sulphate as Standard for Permanganate Solutions, 520
Miller, E. H., Note on the Gravimetric Estimation of Phosphorus in Milk, 579
Mirto, D., Comparative Values of the Biological Reactions with Serum-Precipitin and the so-called Erythro-Precipitin for the Detection of Blood, 590
Mitchell, C. A., *and* Prideaux, R. M., Fibres Used in Textile and Allied Industries, *Review*, 322
————————, *and* Simmons, W. H., Edible Fats and Oils: Their Composition, Manufacture, and Analysis, *Review*, 487
Moeckel, K., *and* Frank, E., Estimation of Sugar in Blood, 73
Monier-Williams, G. W., Note on the Gravimetric Estimation of Phosphorus in Milk, 582
————————————, II., On the Chemical Changes Produced in Flour by Bleaching, 254
Monnier, A., Analysis of Calcium Cyanamide, 429
Monthule, C., Estimation of Theobromine and Caffeine, 501
——————, Estimation of Theobromine and Caffeine in the Presence of Each Other, 297
Mooser, W., Detection of Poisonous Ricinus Constituents in Fodder-Stuffs, 506
Moreau, E., Biological Examination of Honey (Enzymes), 220
—————, *and* Sartory, A., Bacteriological Examination of Honey, 505
————————————, Bacteriological Study of Honey, 353
————, L., *and* Vinet, E., Proportion of Lead Arsenate in the Products of Vines Treated with Arsenical Washes, 301

Morgan, G. T., *and* Cooper, E. A., Germicidal Action of Arsenic and Antimony Compounds on *Bacillus Typhosus*, 282
———, J. L. R., Constant-Temperature Bath for Use at Temperatures both Above and Below that of the Room, 249
———, R. J.. Estimation of Tin in Ores, 527
Moser, L., Estimation of Nitric Oxide, and its Behaviour towards Water, 433
———, *and* Machiedo, L., Separation of Strontium from Calcium, 247
Mott, F. E., Cider Vinegar, 589
Moufang, E., *and* Scheer, A., Valuation of Hops by Analysis, 587
Muer, H. F., Determination of Sulphur in Coal by Means of Jackson's Candle Turbidimeter, 519
Müller, E., *and* Diefenthäler, O., Simultaneous Volumetric Estimation of Iron and Vanadium, 475
———, *and* Diethelm, B., Estimation of Carbon and Sulphur in High-Grade Alloys of Tungsten, Molybdenum, and Vanadium with Iron, 31
———, *and* Wezelin, G., Volumetric Estimation of Ferric Iron by Permanganate after Reduction with Zinc, 521
———, F., *and* Albrecht, A., Estimation of Naphthalene in Crude Coal Gas, 510
———, *and* Thoms, H., Nature of the Poisonous "Cardamon" (Maratti) Fat Employed in the Manufacture of "Backa-Margarine," 542
———, G., *and* Autenrieth, W., Colorimetric Estimation of Sugar, Creatine, and Creatinine in Urine, 591
, J. H., Action of Salicylic Acid on Metallic Acids, 567
Mullikin, S. P., Identification of the Commercial Dyestuffs, *Review*, 385
Munroe, C. E., *and* Hall, C., A Primer on Explosives for Coal-Miners, *Review*, 380
Murrmann, E., Quantitative Estimation of Lithium, 175
———, Separation of Lime from Large Quantities of Magnesia, 242
Myers, J. E., *and* Firth, J. B., Apparatus for Precipitating, Filtering, and Drying in an Inert Gas, 313
Mylius, F., Quantitative Analysis of Gold with the Use of Ether, 303
———, *and* Hüttner, C., Use of Ether in the Analysis of Metals, 431

Nash, L. M., Note on Henry C. Frey's Method of Estimating Petroleum in Turpentine, 138
———, Note on the Examination of Finnish Turpentine, 577
———, The Degree of Accuracy with which Proteins can be Estimated in Milk by Aldehyde Titration, 11
———, *and* Coste, J. H., A Further Contribution to the Question of Turpentine Substitutes, 207
Nelson, E. K., Estimation of Ketones in Essential Oils, 514
Neuberg, C., *and* Saneyoshi, S., Detection of Small Quantities of Disaccharides, 594
———, Detection of Small Quantities of Glycuronic Acid, 596
Neumann, R., Estimation of Fat in Feeding-Stuffs by Means of Trichlorethylene, 542
Ney, N., Kneip, A., *and* Reimers, F., Quantitative Estimation of Cantharidine in Cantharides and in Tinctures, 343
Nicolas, E., On the Peroxydase of Cow's Milk and its Detection by Means of the *p*-phenylenediamine Test, 222
Niederstadt, —, Candelilla Wax, 598
Niegermann, C., Apparatus for the Estimation of the Hexabromide Value of Oils, 607
Nierenstein, M., Quantitative Estimation of Tannin by Means of Casein, 123
Nierstein, M., *and* Daniel, K. C. R., Use of Carbalkyloxy Derivatives for the Quantitative Estimation of Hydroxyl Groups, 227
Nockmann, E., Detection of Cocoanut Oil in Butter by Means of Ewer's Method, 451

Nott, H., Estimation of Organic Substances in Water by Means of Permanganate, 502
Nottbohm, F. E., *and* Angerhausen, J., Wendler's Method for the Estimation of Fat in Milk, 14
Nowicki, R., Carbon Monoxide Detector, 608
Nydegger, O., Estimation of Chromium in Chrome Ironstone, 428
Nyman, M., *and* Bjorksten, R., Precipitation of Cocaine Solutions with Platinum Chloride, 354

Oddo, B., Estimation of Active Hydrogen in Organic Molecules, 459
Odell, A. F., Modified Boltwood Vacuum Pump, 128
Oettinger, C., *and* Buchta, F., Soya Bean Oil, 358
Ogilvie, J. P., Determination of Sucrose in Beet Sugar Factory Products by Clerget's Process, Using Invertase as Hydrolyst, 120
Oppler, B., Estimation of Chlorine in Blood, 72
Ordonneau, C., Estimation of Total Tartaric Acid by Goldenberg's Method, 81
Orton, K. J. P., *and* Edwards, M. G., Detection and Estimation of Small Quantities of Acetic Anhydride in Acetic Acid, 417
Osterberg, E., *and* Wolf, C. G. L., Estimation of Sulphur and Phosphorus in Organic Substances, 81
O'Sullivan, J., Improvement in the Method of Malt Analysis, 146
Outerbridge, A. E., Junr., Detection of Hydrocarbon and Resin Oils in other Oils, 513

Parkes, A. E., Note on Gorgonzola Cheese, 63
————, *and* Roberts, J. D., Note on the Composition of Blaud's Pill, 389
————————, Note on the "Pearl Coating" of Pills, 389
Parry, J. E., Analysis of Liquorice Juice, 410
Passerini, N., Colorimetric Method of Estimating Phosphoric Acid, 307
Patrick, W. A., *and* Walker, W. H., Method for Testing Galvanised Iron to Replace the Preece Test, 304
Patterson, G. W., Stability Tests of Smokeless Powder, 295
Peckham, S. F., Practical Value of Calorimetry in Testing Anthracite, 562
Pellet, H., Estimation of Nitric Nitrogen in Gun-Cotton, Nitroglycerine, and the Like, 513
Pence, F. K., Determination of Hydrated Silicic Acid in Clay, 85
Pépin, G., Physical Constants and Analyses of Certain Commercial Peptones, 111
Perdue, W. H., *and* Hulett, G. A., Exact Method for the Electrolytic Estimation of Metals, 370
Péron, G, *and* Bernier, R., Estimation of Small Quantities of Iodides in the Presence of Other Compounds, 241
Philip, A., *and* Steele, L. J., New Type of Automatic Inflammable Gas and Vapour Detector, 528
————, *and* Stenhouse, J., Methods of Testing Safety Lamps and other Apparatus for the Detection of Combustible Gases and Vapour, 528
Philippe, E., Applicability of Recent Methods for the Examination of Milk, 347
————, *and* Schaffer, F., Examination of Absinthe and its Imitations, 272
Phillips, P. P., Gum from *Bombax Malabricum*, 356
Pickles, S. S., *and* Hayworth, W. P., Composition of Para Rubber-Seed Oil, 491
————————, Composition of the Fat from the Seeds of *Lophira alata* (Banks), 493
Pighini, G., Estimation of the Enzymatic Activity of Nuclease, 109
Pilch, F., Volumetric Analysis with Small Volumes of Liquid, 185
Pinnow, J., Estimation of Hydroquinone, 163

Plucker, W., Cause of the Poisonous Properties of Certain Brands of German Margarine, 216
Poda, —, Thermostat for Refractometric Examinations, 89
Poeschl, V., An Introduction to the Chemistry of Colloids, *Review*, 485
Polenske, E., Detection of Benzoic Acid in Foods, etc., 584
————, Estimation of Small Quantities of Water in Lard, 497
————, *and* Baur, E., Method for the Separation of Starch and Glycogen, 501
————, *and* Köpke, O., Estimation of Potassium Nitrate in Meat, 217
Pollock, C., *and* Benson, H. K., New Accelerated Test for Paints, 608
Pope, G. T., Purchase of Coal under Specifications, 229
Pouget, I., *and* Chouchak, D., Colorimetric Estimation of Phosphoric Acid, 525
Powney, W. E. F., *and* Coste, J. H., Testing of Incandescent Mantles, 125
Pozzi-Escot, E., Rapid Qualitative Detection of Elements, the Sulphides of which are Precipitated by Hydrogen Sulphide in Acid Solution, 568
————, Use of Dimethylaminobenzene-azobenzene Sulphonic Acid as a Reagent in Microchemistry, 232
Pratt, D. S., Chamot, E. M., *and* Redfield, H. W., Study of the Phenolsulphonic Acid Method for the Estimation of Nitrates in Water, III., Chief Sources of Error, 244
————————————, Study of the Phenolsulphonic Acid Method for the Estimation of Nitrates in Water, IV., A Modified Method, 244
——, L. A., *and* James, C., New Method for Separation of Cerium, 518
Price, T. S., *and* Hyde, A. W. T., Adaptation of the Tap-funnel to Rapid Electro-Analysis with Stationary Electrodes, 313
Prideaux, B. M., *and* Mitchell, C. A., Fibres Used in Textile and Allied Industries, *Review*, 322
Probeck, E., Estimation of Glycerol, 289
Procter, H. R. *and* Seymour-Jones, R. A., Estimation of Soluble Mercuric Salts at Great Dilutions, 305
Puran Singh, Analytical Constants of Shellac, Lac-Resin, and Lac-Wax, 119

Quadrat, O., *and* Milbauer, J., Ferric Sulphate as Standard for Permanganate Solutions, 520
Quartaroli, A., Rapid Method for the Estimation of Nitrates, 468
Quincke, G. A., A New Extraction Apparatus, 529

Race, J., *and* Ross, R., Constants of Chicken and Turkey Fats, 213
————————, Note on Almond and Apricot Kernel Oils, 263
————————, *and* Maudsley, F., Examination of Shrewsbury and Knapp's Process for the Estimation of Cocoanut Oil, 195
Raiford, L. C., *and* Heyl, F. W., Analysis of *Zygadenus Intermedius* ("Death Camas") 156
Raikow, P. N., *and* Tischkow, P., Estimation of Manganese as Oxide, 566
Raquet, D., *and* Caron, H., Examination of Bismuth Salicylate, 342
Rather, J. B., Estimating Humus in Soils, 597
Rathgen, F., Extinguishing the Flame of Petroleum Spirit, 252
Raulin, G., Rapid Estimation of Nickel in Steel, 373
Ray, B. J., *and* Withers, W. A., Modification of the Diphenylamine Test for Nitrous and Nitric Acids, 370
Reckleben, H., *and* Scheiber, J., Attempts to Prepare a Solid Hydride of Antimony, 300
————————————, Detection of the Solid Hydride of Arsenic, 300

Redfield, H. W., Chamot, E. M., *and* Pratt, D. S., Study of the Phenolsulphonic Acid Method for the Estimation of Nitrates in Water, III., Chief Sources of Error, 244
———————————————, Study of the Phenolsulphonic Acid Method for the Estimation of Nitrates in Water, IV., A Modified Method, 244
Reich, R., Composition of Ripe and Unripe Bananas, 541
Reichard, C., Glucoside Reactions: Convallarin and Convallamarin, 289
Reimers, F., Kneip, A., *and* Ney, N., Quantitative Estimation of Cantharidine in Cantharides and in Tinctures, 343
Reinhardt, R., *and* Siebold, E., Behaviour of Schardinger's Reagent towards Colostrum, 221
Reinthaler, F., Volumetric Method for the Estimation of Mercury Depending on Reduction to Metal, 431
Remmier, H., Absorption of Arsenic by Sugar Beets, 549
Remy, T., *and* Rösing, G., Bacteriological Investigation of Soil, 219
Reuss, A., *and* Heiduschka, A., Estimation of Arsenic in Schweinfurt Green, 363
Revis, C., Note on the Gravimetric Estimation of Phosphorus in Milk, 582
————, The Composition of Milk, 392
————, The Degree of Accuracy with which Proteins can be Estimated in Milk by Aldehyde Titration, 11
————, *and* Bolton, E. R., Further Analyses of Ghee, 392
——————————, Observations on Some Methods of Estimating Cocoanut Oil and Butter in Butter and Margarine, 333
Ribaut, H., Estimation of Aconitine by Silicotungstic Acid, 142
Richards, P. A. E., Note on Gorgonzola Cheese, 63
————————, *and* Cribb, C. H., Notes on the Analysis of Margarine, 327
Richardson, F. W., A Further Contribution to the Question of Turpentine Substitutes, 212
————————, Note on the Examination of Finnish Turpentine, 578
————————, The Analytical and Microscopical Examination of Compound Liquorice Powder, 202
————————, *and* Whitaker, J. E. F., Analysis of Oils of Turpentine, II., 150
Richmond, H. D., Note on Gorgonzola Cheese, 63
————————, Note on the Gravimetric Estimation of Phosphorus in Milk, 583
————————, Observations on Some Methods of Estimating Cocoanut Oil and Butter in Butter and Margarine, 340
————————, The Composition of Milk, 390
————————, The Degree of Accuracy with which Proteins can be Estimated in Milk by Aldehyde Titration, 9
————————, The "Neusal" Milk Test, 206
Richter, E., *and* Lenz, W., Detection of Perborates and Similar Compounds, 470
Roberts, E. J., Separation of Cerium by Potassium Permanganate, 429
————, J. D., *and* Parkes, A. E., Note on the Composition of Blaud's Pill, 387
——————————————, Note on the "Pearl Coating" of Pills, 389
Robinson, R. A., Bell, W. J., *and* Lloyd, C. F., The Sale of Food and Drugs Acts, *Review*, 325
Rockwood, E. W., Digestibility of Bleached Flour, 64
Roettgen, T., *and* Windisch, K., Estimation of the Volatile Acids in Wine, 502
Rogers, A., *and* Sabin, A. H., Consistency of Paints by the Stormer Viscosimeter, 609
Röhrig, A., Estimation of Total Fatty Acids in Soap, 20
Romijn, G., Estimation of Nitrate, 467
Ronnet, L., Analysis of Commercial Absinthe, 12
————, Simultaneous Estimation of Ferrocyanides and Thiocyanates, 563

Rosenheim, A., *and* Weinheber, M., Estimation of Tellurium, 179

——————————, W., Gravimetric Estimation of Tellurium and Alkalimetric Estimation of Telluric Acid, 248

Rosenthaler, L., Hydrargyrometric Analyses, 357

————, *and* Schaeffer, W., Composition of Sweet Apricot Kernels, 450

Rosin, J., Volumetric Estimation of Phosphoric Acid, 472

Rösing, G., *and* Remy, T., Bacteriological Investigation of Soil, 219

Ross, W. H., *and* Benner, R. C., Rapid Determination of Silver and Cadmium by Means of the Gauze Cathode and Stationary Anode, 473

——————————, Rapid Determination of Nickel and Cobalt by Means of the Gauze Cathode and Stationary Anode, 307

——, R., *and* Race, J., Constants of Chicken and Turkey Fats, 213

——————, Note on Almond and Apricot Kernel Oils, 263

——, *and* Maudsley, F., Examination of Shrewsbury and Knapp's Process for the Estimation of Cocoanut Oil, 195

Rossiter, E. C., Estimation of Sulphides in Alkali-Metal Cyanides, 436

Rössler, L., Estimation of Gold by Means of Hydrogen Peroxide, 34

Rothberg, P., *and* Curtman, L. J., Application of the "Glow Reaction" to the Detection of the Platinum Metals, 434

——————————, Efficiency of Borax Bead Tests for Nickel and Cobalt in Admixture, 173

Roush, G. A., Microscopical Examination and Identification of Carbon, 428

Ruff, O., *and* Gersten, E., Some Sources of Error in Gasometric Estimation of Nitrate by Schloesing's Method and of Nitrite by Piccini's Method, 525

Rupp, E., Phenolphthaleïn Derivatives and their Properties as Indicators, 169

———— *and* Kroll, —, Analysis of Calcium Hypophosphite, 605

———— *and* Lehmann, F., Decomposition of Silicates by Fusion with Boric Acid as Applied to Qualitative Analysis, 371

——————————, New Method of Estimating Nitrites, 307

——————————, Simplified Method for the Estimation of Arsenic in Atoxyl and Arsacetin, 273

Russell, E., *and* Hodgson, T. R., Note on the Composition of British Wines, 60

Rutherford, T. A., Howe, P. E., *and* Hawk, P. B., Preservation of Fæces, 74

Sabin, A. H., *and* Rogers, A., Consistency of Paints by the Stormer Viscosimeter, 609

Sacher, J. F., Sensitive Indicator, 26

————, Volumetric Estimation of Lead Peroxide, 467

Sage, C. E., Testing of Coal-Tar Creosote, 419

Saint-Sernin, A., Biological Method for Identifying Various Fleshes, 455

Salkowski, E., Detection of Mercury in Urine, 552

Sanchez, J. A., New Method of Separating Iron from Manganese, 604

Sand, H. J. S., Apparatus for Rapid Electro-Analytical Determination of Metals, 181

————, *and* Law, D. J., Electrometric Method for the Estimation of the Acidity of Tan Liquors, 121

——, H. J. S., Wood, J. T., *and* Law, D. J., Electrometric Method for the Estimation of the Acidity of Tan Liquors. Part II., 515

Saneyoshi, S., *and* Neuberg, C., Detection of Small Quantities of Disaccharides, 594

——————————, Detection of Small Quantities of Glycuronic Acid, 596

Sartory, A., *and* Moran, C., Bacteriological Examination of Honey, 505

——————————, Bacteriological Study of Honey, 353

Sasserath, A., Moroccan Olive Oil, 107

Scandola, E., Colorimetric Estimation of Strychnine, 144

Scarafia, —, *and* Francesconi, L., Essential Oil of *Santolina Chamæcyparissus*, 589

Schacht, G., Testing of Imitation Parchment Papers for Resistance to Grease, 597

Schaeffer, W., *and* Rosenthaler, L., Composition of Sweet Apricot Kernels, 450
Schaffer, F., Detection of Invertase in Wine, 350
——————, *and* Philippe, E., Examination of Absinthe and its Imitations, 272
Schaller, W. T., Bismuth Ochres from California, 174
Scheer, A., *and* Moufang, E., Valuation of Hops by Analysis, 587
Scheiber, J., *and* Reckleben, H., Attempts to Prepare a Solid Hydride of Antimony, 300
——————————————, Detection of the Solid Hydride of Arsenic, 300
Scheitz, P., The Alcohol-Soluble Constituents of Litmus, 25
Scheringa, K., Estimation of Potassium Perchlorate in Potassium Chlorate, 248
Schidrowitz, P., *and* Goldsbrough, H. A., Rubber, *Review*, 532
——————————————————, Note on the Detection and Estimation of Small Quantities of Antimony, 101
Schiffer, P., *and* Strecker, W., Titration of Phosphates, 473
Schirm, E., Precipitation of Aluminium, Chromium, and Iron by Ammonium Nitrite, 559
—————, Precipitation of Iron with Hydrazine Hydrate, 521
—————, Precipitation of Zinc, Manganese, Cobalt, Nickel, Copper, and Cadmium, from Ammoniacal Solutions by Means of Sodium Carbonate and Trimethylphenyl-ammonium Carbonate, 599
—————, Separation of Zinc and Sulphuric Acid, 607
Schlüter, H., *and* Schwarz, F., Lubricating Oils for Motor-Cars and Gas-Engines, 291
Schmidt, H., *and* Abderhalden, E., Use of Triketohydrindene Hydrate for the Detection of Proteins and their Derivatives, 514
Scholl, A., Greifenhagen, W., *and* König, J., Estimation of Gelatin, 585
————————————————————, Estimation of Starch, 588
Scholz, A., *and* Lührig, H., Examination of Honey by Fiehe's Reaction, 451
Schoorl, N., Microchemical Test for Aluminium by Means of Caesium Chloride, 362
——————, *and* Lenz, W., Microchemical Test for Sodium, 372
Schreiber, H., *and* Taber, W. C., Estimation of Tin in Canned Foods, 149
Schreiner, O., *and* Lathrop, E., Dihydroxystearic Acid in Good and Poor Soils, 504
Schröder, J., Estimation of Nicotine in Concentrated Tobacco-Juice, 106
——————, Tobacco Extracts and their Value, 411
Schroeder, K., Preparation of Pure Potassium Ferrocyanide and its Analytical Control, 564
Schröter, F., Estimation of Hexamethylenetetramine (Urotropine) in Urine, 285
Schubert, H., *and* Knorr, L., Colorimetric Method for the Quantitative Estimation of Enols in Allelotropic Mixtures, 554
(Schück, B.), *and* Caro, N., Analysis of Commercial Calcium Cyanamide, 76
Schwarz, F., *and* Schüter, H., Lubricating Oils for Motor-Cars and Gas-Engines, 291
Schwert, W., Beythien, A., Hempel, H., Simmich, P., *and* Wiesemann, Estimation of Formaldehyde by the Ammonia Method, 421
Scott, J. H., Spence, D., *and* Galletly, J. C., Estimation of Rubber as Tetrabromide. Decomposition of the Bromide by Nitric Acid, 361
Scott-Smith, J. E., *and* Evans, J., The Analytical and Microscopical Examination of Compound Liquorice Powder, 198
Scott-Wilson, H., Method for the Estimation of Acetone in Animal Fluids, 548
Scoville, W. L., Estimation of Minute Quantities of Nitroglycerin, 556
Sebelien, J., *and* Krog, K., Quantitative Estimation of Nitric Acid in Vegetable Products, 163
Seeker, A. F., *and* Mathewson, W. E., Estimation of Iodine in Organic Compounds, and its Separation from other Halogens, 118
Seiter, F. J., *and* Enger, F., Identification of Cocaine and Substitutes, 405
Semmler, F. W., *and* Zaar, B., Essential Oil of "False Camphor-Wood." The Occurrence of Myrtenal and *d*-Perilla Aldehyde in Nature, 289

Sen, H. K., *and* Dey, B. B., Action of Hydrazine Sulphate on Nitrites, and a Method for the Estimation of Nitrite Nitrogen, 468
Serger, H., Colour Reactions of Edible Fats and Oils, 406
Seymour-Jones, R. A., *and* Procter, H. R., Estimation of Soluble Mercuric Salts at Great Dilutions, 305
Shadinger, G. B., *and* Brown, C. E., Flask for Distillation of Tar, 529
Shen, B., *and* Findlay, A., Solubility of Carbon Dioxide in Beer, 404
Sherman, H. C., *and* Gross, A., Detection of Salicylic Acid, 463
Shippen, A. E., *and* Gill, A. H., Estimation of Unsaponifiable Matter in Wool Oils, 168
Shrewsbury, H. T., Note on Henry C. Frey's Method of Estimating Petroleum in Turpentine, 137
Siau, R. L., New Form of Constant Temperature Drying-Oven, 126
Siebold, E., *and* Reinhardt, R., Behaviour of Schardinger's Reagent towards Colostrum, 221
Siegfried, M., *and* Zimmermann, R., Estimation of Phenol and *p*-Cresol in the Presence of Each Other, 79
Silber, J. M., Estimation of the Hardness of Water: the Wartha-Pfeiffer Method and its Modification, 311
Silberrad, O., Improved Soxhlet Condenser, 528
Silberstein, S., *and* Bardach, B., Polarimetric Estimation of Sugar by the Method of Jolles, 361
Silva, F. da, Preparation of Madeira and its Composition, 410
Simmich, P., Estimation of the Total Fatty Acids in Fat, Oils, and Soaps, 115
——————, Beythien, A., Hempel, H., Schwert, W., *and* Wiesemann, C., Estimation of Formaldehyde by the Ammonia Method, 421
Simmons, W. H., *and* Mitchell, C. A., Edible Fats and Oils: Their Composition, Manufacture, and Analysis, *Review*, 487
Simonot, E., Rapid Gravimetric Method for the Estimation of Albumin in Urine, 551
Simonson, J. L., Some Reactions of Gum Kino, 544
Singh, P., Use of Nickel Hydroxide in Tannin Estimation, 516
Slator, A., Gas Regulator for Thermostats, 127
Slemssen, J. A., Reaction for Uranium Salts, 180
Slyke, D. D. van, Estimation of Amino Groups in Amino Compounds and in Urine, and a Method for the Analysis of Proteins, 457
————————, Quantitative Estimation of Aliphatic Amino Groups, and its Application in the Chemistry of the Proteins, Urine, and Enzymes, 17
Smalley, W. U., Electrolytic Estimation of Copper and Zinc, Using Sand's Apparatus, 182
Smith, A., *and* Menzies, A. W. C., Static Method for Determining Vapour Pressures of Solids and Liquids, 36
———, E. F., *and* Buckminster, I. H., Electrolytic Separations, 32
——————, *and* Goldbaum, J. S., Electrolytic Determination of Chlorine in Hydrochloric Acid with the Use of a Silver Anode and Mercury Cathode, 43
———, F., *and* Brünnich, J. C., Detection and Estimation of Arsenic Acid in Presence of Arsenious Acid by Means of Magnesia Mixture, 30
———, L. H., Some Constants of Mutton-Bird Oil, 290
Sobbe, O. von, Control Tests of the Colour Scale Used with Schaeffer's Curd Tester, 378
Sobolewa, W., *and* Zaleski, J., Quantitative Estimation of Acetaldehyde by Means of Pyrrol, 157
Solling, J., *and* Hartel, F., Analysis and Composition of Jams, 144
Sornet, R., *and* Delépine, M., Separation and Estimation of Ammonia and Pyridine, 508

Soukup, A., *and* Hanŭs, J., Estimation of Copper by Means of Hypophosphorous Acid, 302
Southcombe, J. E., Deposits Resulting from the Lubrication of Engines, 230
Spence, D., *and* Galletly, J. C., Estimation of Caoutchouc as Tetrabromide, 509
———, Galletly, J. C., *and* Scott, J. H., Estimation of Rubber as Tetrabromide. Decomposition of the Bromide by Nitric Acid, 361
Spengel, A., *and* Wöhler, L., Separation of Platinum and Tin, 177
Splittgerber, A., *and* Tillmans, J., Estimation of Nitrates in Milk by Means of the Diphenylamine-Sulphuric Acid Test, 587
Spring, La V. W., Method for the Separation of Nickel and Zinc in German Silver and Other Alloys, 524
Spurrier, H., Prevention of Bumping, 608
———, Rapid Filtering Funnel, 609
Stanck, V., Detection of Chromium in Steel, 428
Stansfield, E., Simple Forms of Gas-Pressure Regulators, 477
Starck, G., Estimation of Fluorine, 303
Steinkopf, W., Apparatus for Filtering Hygroscopic Substances or those affected by Air, 87
———, Apparatus for Storing and Measuring Poisonous, Hygroscopic, or Volatile Liquids, 88
Steinmann, A., Assay of Platinum, 605
———, Assay of Silver by Means of the Touchstone, 372
Stenhouse, T., *and* Philip, A., Methods of Testing Safety Lamps or Other Apparatus for the Detection of Combustible Gases and Vapour, 528
Starkenstein, E., Behaviour of Uranium Phosphates towards Indicators, 375
Stecher, E., Fischer, F., *and* Thiele, C., Rapid Electro-Analysis with Agitation by Means of a Current of Gas, 600
———, Rapid Electro-Analysis under Reduced Pressure, 601
Steele, L. J., *and* Philip, A., New Type of Automatic Inflammable Gas and Vapour Detector, 528
Steppuhn, O., *and* Gottlieb, R., Quantitative Estimation of Morphine, 142
Stevens, A. B., Assay of Aconite, 450
———, H. P., *and* Beadle, C., Rubber, *Review*, 443
———, Some Analyses of Hevea Latex, 6
Stevenson, R., *and* Baskerville, C., Analytical Examination of Commercial Oxygen, 469
———, Chemistry of Anæsthetics. III. Nitrous Oxide, 503
———, Oxidation of Ferrous Salts, 467
Stœcklin, L., Cryoscopic Examination of Milk, 345
Strachan, J., "Hughes' Reaction" of Potassium Iodide on Paper, and its Bearing on the Question of Acidity, 292
Straughn, M. N., Bryan, A. H., *and* Given, A., Estimation of Sugars in Feeding-Stuffs, 453
Strecker, F., Combined Fixing and Staining Processes for Microscopical Preparations, 569
———, W., *and* Schiffer, P., Titration of Phosphates, 473
Stutzer, A., Analysis of Calcium Cyanamide, 429
———, —, *and* Goy, —, Estimation of Nitrate and Nitrite Nitrogen in Commercial Calcium Nitrate and Nitrite, 524
Surre, L., Estimation of Nicotine in the Presence of Pyridine Bases, 452
Susuki, S., Occurrence, Detection, and Estimation of Nitrous Oxide in the Gases Produced by Denitrifying Organisms, 591

Sutthoff, W., *and* Tillmans, J., Detection and Estimation of Nitric Acid and Nitrous Acid in Water, 433
Sutton, F., A Systematic Handbook of Volumetric Analysis, *Review*, 484
Szilard, B., Apparatus for Measuring Radioactivity, 380

Taber, W. C., *and* Schreiber, H., Estimation of Tin in Canned Foods, 149
Tangl, F., *and* Von Kereszty, G., Wet Method for the Estimation of Carbon in Organic Substances, 354
Tassilly, E., *and* Leroide, J., Relative Proportions of Arsenic in Marine Algæ and Products Derived from Them, 218
Taurel, —, *and* Griffett, —, Estimation of Sublimed Sulphur in a Mixture of Different Kinds of Sulphur, 374
Taylor, A. E., Sources of Error in the Folin Method of Estimating Creatinine, 288
———, W. E., Precipitation of Aluminium Hydroxide in Granular Form, 229
Thiele, C., Fischer, F., *and* Stecher, E., Rapid Electro-Analysis with Agitation by Means of a Current of Gas, 600
———————————————, Rapid Electro-Analysis under Reduced Pressure, 601
Thian, J., *and* Hanůs, J., Estimation of Cocoanut Oil in Lard by Means of the "Ethyl Ester Value," 106
Thoms, H., and Müller, F., Nature of the Poisonous "Cardamon" (Maratti) Fat Employed in the Manufacture of "Backa-Margarine," 542
Thomson, J. J., New Method of Chemical Analysis, 376
———, W., Estimation of Indigotin in the Presence of Starch, 290
Thöni, J., Use of the Quantitative "Precipitin" Reaction in Honey Analysis, 456
Thorburn, A. D., Estimation of Morphine by Extraction with Phenyl-ethyl Carbinol, 587
Tillmans, J., Detection and Estimation of Nitrates in Milk by Means of the Diphenylamine-Sulphuric Acid Test, 67
————, *and* Henblein, O., Titration of Alkali Carbonates in Presence of Alkali Hydroxides or Bicarbonates, 362
————, *and* Splittgerber, A., Estimation of Nitrates by Means of the Diphenylamine-Sulphuric Acid Test, 587
————, *and* Sutthoff, W., Detection and Estimation of Nitric Acid and Nitrous Acid in Water, 433
Tizard, H. T., Methyl Red as an Indicator Compared with Methyl Orange, 118
Tischkow, P., *and* Raikow, P. N., Estimation of Manganese as Oxide, 566
Tollens, B., *and* Böddener, K. H., Modification of the Furfural Method for the Estimation of Pentosans, 161
————, *and* Ishida, M,, Estimation of Pentosans and Methylpentosans in Cereals and Wood Fungi, 460
————, *and* Wichers, Pentosans of Certain Wood Fungi, 162
Torquati, J., Estimation of Hordenine in Germinating Barley, 162
Touplain, F., *and* Bordas, F., Distribution of the Phosphoric Acid in Milk, 345
Touzalin, L. A., *and* Brady, W., Determination of Dust in Blast Furnace Gas, 601
Traube, J., Capillary Analysis, 251
Trautmann, W., Analysis of Ferrotitanium Alloys Containing Much Silicon, 373
—————, Analysis of Ferro-Uranium, 125
—————, Analysis of Ferro-Vanadium and Estimation of Vanadium in Presence of Arsenic, 437
—————, Analysis of Ferro-Zircon, 124
—————, Analysis of Molybdenum Glance, 243
—————, Estimation of Aluminium and Chromium in Ferro-vanadium, 123

Trautmann, W., Estimation of Silicon in Vanadium and Molybdenum and in their Iron Alloys, 311

Treadwell, F. P., Analytical Chemistry, *Review*, 611

————, W. D., Titration of Potassium Cyanide in Presence of Potassium Ferrocyanide, 460

Tsujimoto, M., Rice Oil, 357

Tyrer, D., Method for the Accurate Estimation of Traces of Water in Ether, 420

Ulex, H., Estimation of Nicotine in Concentrated Tobacco Juices, 143

Utescher, —, Analysis of Fulminate Cap-Compositions, 595

Utz, K., Niger-Seed Oil, 358

Van den Bos, J. L. M. van der Horn, Quantitative Separation of Barium, Strontium, and Calcium, 364

—— Slyke, D. D., Estimation of Amino Groups in Amino Compounds and in Urine, and a Method for the Analysis of Proteins, 457

————————, Quantitative Estimation of Aliphatic Amino Groups, and its Application in the Chemistry of the Proteins, Urine, and Enzymes, 17

Vandevelde, A. J. J., Experiments on the Precipitation of Proteins, 295

Vassals, E., Use of Logwood in Qualitative Analysis. Estimation of Bismuth, 603

Vaubel, W., Estimation of the Bromine Value of Fats and Oils. Primary and Secondary Bromine Values, 19

Vauflart, L., Detection and Estimation of Calcium Cyanamide in the Presence of Other Fertilisers, 466

Veley, V. H., Use of Indicators for Determing Affinity Value, 569

Vernon, H. M., Estimation of Indophenol Oxydase of Animal Tissues, 550

Vierhout, P., Estimation of Salicylic Acid in Fruit Juices, 411

Vinet, E., *and* Moreau, L., Proportion of Lead Arsenate in the Products of Vines Treated with Arsenical Washes, 301

Virchow, C., Estimation of Lecithin, 510

Vittoria, E., Improved Hollemann's Method for the Estimation of Reverted Phosphoric Acid, 605

Voelcker, E. W., Observations on Some Methods of Estimating Cocoanut Oil and Butter in Butter and Margarine, 340

Vogtherr, M., Hæmoglobin Preparations, 222

Vollant, F., Estimation of Sucrose and the Detection of Dextrin in Foods, 588

Von Degrazia, J., New Method for the Estimation of Nicotine in Tobacco, 280

————————, Polarimetric Estimation of Nicotine in Tobacco Extracts, 279

Von der Heide, R., Condenser and Extraction Apparatus, 378

Von Fellenberg, J., Colour Reactions of Aromatic Aldehydes and their Applicability in the Analysis of Spirits, 273

————————, Higher Alcohols Occurring in Jamaica Rum, 411

————————, Detection of Resin in Glazed Coffee, 275

Von Fenyvessy, B., Influence of Certain Vegetable Proteins on Wheat Gluten, 407

Von Fürth, O., Detection of Blood, 549

Von Huber, H., *and* Marcusson, J., Detection of Train Oil in Oils, Fats, and Soaps, 359

Von Kereszty, G., *and* Tangl, F., Wet Method for the Estimation of Carbon in Organic Substances, 354

Von Liebermann, L., Apparatus for the Estimation of the Melting-Point of Fats, 569

————————, Laboratory Apparatus for the Production of a Constant Current of Ozonised Air, 479

————————, *and* Andriska, V., Method of Estimating the Quality of Wheaten Flour, 543

Von Sobbe, O., Control Tests of the Colour Scale Used with Schaeffer's Curd Tester, 378
Voorhoeve, N., Method for the Estimation of Small Variations in the Quantity of Calcium Contained in Blood, 220

Wacker, L., Colorimetric Method for the Estimation of the Molecular Weight of Polysaccharides, 360
Waddell, J., Volumetric Determination of Lead as Chromate, 602
Wade, J., *and* Merriman, R. W., Apparatus for the Maintenance of Constant Pressures above and below the Atmospheric Pressure. Application to Fractional Distillation, 441
Wagenaar, M., Detection of Fat in Beeswax, Paraffin, Spermaceti, etc., 496
Walden, A. E., *and* Lander, G. D., The Detection of Traces of Hydrogen Cyanide, 266
Walker, W. H., *and* Patrick, W. A., Method for Testing Galvanised Iron to Replace the Preece Test, 304
Walpole, G. S., Direct Estimation of Creatine in Pathological Urine, 504
Warcollier, G., Estimation of Tartaric Acid in Apples, Pears, Cider, and Perry, 545
Ward, G. B., *and* Brown, A. J., Valuation of the Antiseptic Properties of Hops, 65
Warren, W. H., Detection of Gas in Sealed Tube Reactions, 531
Warunis, J. S., Estimation of Halogens in Organic Compounds, 509
———, Estimation of Sulphur in Organic Compounds, 80
———, Estimation of Total Sulphur in Coal, 375
Waters, C. E., Potash Bulb, 86
Watson, H. E., Volumetric Estimation of Oxygen in Air, 569
Wauters, M. J., Note on the Detection of Cocoanut Oil in Chocolate, 275
Wdowiszewski, H., Estimation of Carborundum in Coke Crucible Scrap, 31
———, *and* Boguluboff, P., Estimation of Chromium in Chrome Tungsten Steel, 33
Wechsler, E., Phosphotungstic Acid Precipitation of Proteins, 461
Wegelin, G., *and* Müller, E., Volumetric Estimation of Ferric Iron by Permanganate after Reduction with Zinc, 521
Weil, H., New Reaction for the Differentiation of Cobalt and Nickel, 238
Weinheber, M., *and* Rosenheim, A., Estimation of Tellurium, 179
———, Gravimetric Estimation of Tellurium and Alkalimetric Estimation of Telluric Acid, 248
Weisz, M., Relation of the Normal Yellow Pigmentary Substances of Urine to Diazo-Reaction, and a Colorimetric Method for Estimating Urochrome and Urochromogen, 223
Wells, R. C., Sensitiveness of the Colorimetric Estimation of Titanium, 310
Welsh, D. A., *and* Chapman, H. G., Precipitin Reaction, 456
Wender, N., Influence of Inactive Substances on the Rotation of Lævulose, 228
Westcott, W. W., *and* Martindale, W. H., The Extra Pharmacopœia, *Review*, 38
Whitaker, J. E. F., A Further Contribution to the Question of Turpentine Substitutes, 212
———, *and* Richardson, F. W., Analysis of Oils of Turpentine, II., 150
White, F. S., *and* Langmuir, A. C., Analysis of Shellac and Shellac Varnish, 463
Whittier, A. C., Estimation of Inorganic Phosphorus in Animal Tissues, 284
Wichers, J. S., *and* Tollens, B., Pentosans of Certain Wood Fungi, 162
Wiesemann, C., Beythien, A., Hempel, H., Simmich, P., *and* Schwert, W., Estimation of Formaldehyde by the Ammonia Method, 421
Wilhelmi, A., Apparatus for Exact Gas Analysis, 379
Wilkie, J. M., Action of Iodine on Phenols and their Volumetric Determination (Part I.), 294

Wilkie, J. M., Sensitive Test for Phenol and Salicylic Acid, 295
Willcox, —, A Reaction for Caulophyllin, 271
———, The Detection of Traces of Hydrogen Cyanide, 268
Windisch, K., *and* Roettgen, J., Estimation of the Volatile Acids in Wine, 502
Windisch, R., A Casein Product, 406
Winkler, L. W., Estimation of Dissolved Oxygen, 245
———, Estimation of the Saponification Value of Oils, Fats, and Waxes. Use of Propyl Alcohol, 290
Winterfeld, G., Detection of Rosin Oils in French Wool-Grease Oleines, 30
———, *and* Mecklenburg, W., Detection of Mineral Oil in Wool-Grease Oleine, 29
Withers, W. A., *and* Ray, B. J., Modification of the Diphenylamine Test for Nitrous and Nitric Acids, 370
Witte, H., Analysis of Honey, 276
Wöhler, L., *and* Frey, W., Estimation of Acidity in Hydrogen Peroxide Solutions, 82
———, *and* Spengel, A., Separation of Platinum and Tin, 177
Wolf, C. G. L., *and* Osterberg, E., Estimation of Sulphur and Phosphorus in Organic Substances, 81
Wolff, H., Estimation of Ether and Benzene in Alcohol, 19
Wood, J. T., Sand, H. J. S., *and* Law, D. J., Electrometric Method for the Estimation of the Acidity of Tan Liquors, Part II., 515
———, T. B., A Course of Practical Work in Agricultural Chemistry for Senior Students, *Review*, 324
Woy, R., Customs Examimation of Rye and Wheat Bran, 350
Wunder, —, *and* Chéladzé, N., Separation of Aluminium from Beryllium, 426
Wuyts, L., Volumetric Estimation of Phosphoric Acid Soluble in 2 per cent. Citric Acid Solution, 308

Yoder, P. A., Determination of Acids in Sugar-Cane Juice, 594
———, Polarimetric Method for the Determination of Malic Acid, and its Application in Cane and Maple Products, 498
Yoshimura, K., Composition of Ripe and Unripe Bananas, 274

Zaar, B., *and* Semmler, F. W., Essential Oil of "False Camphor-Wood." The Occurrence of Myrtenal and *d*-Perilla Aldehyde in Nature, 289
Zaleski, J., *and* Sobolewa, W., Quantitative Estimation of Acetaldehyde by Means of Pyrrol, 157
Zdarek, E., Distribution of Fluorine in the Human Body, 75
Zenghelis, C., Delicate Test for Hydrogen, 34
Zerewitinoff, T., Quantitative Estimation of Active Hydrogen in Organic Compounds by Means of Magnesium-Methyl Iodide, 117
Zimmermann, R., *and* Siegfried, M., Estimation of Phenol and *p*-Cresol in the Presence of Each Other, 79
Zotier, V., Volumetric Estimation of Phenolphthaleïn, 14

INDEX OF SUBJECTS.

ABSINTHE and its Imitations, Examination of, 272
————, Commercial, Analysis of, 12
————, Thujone in, Detection of, 495
ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS, 12, 64, 103, 142, 214, 272, 342, 404, 450, 495, 541, 584
Accuracy with which Proteins can be Estimated in Milk by Aldehyde Titration, the Degree of, 9
Acetaldehyde, Quantitative Estimation of, by Means of Pyrrol, 157
Acetic Anhydride, Small Quantities of, in Acetic Acid, Detection and Estimation of, 417
Acetin Process for Glycerol Determination, Report on, 316
Acetone, Alcohol in the Presence of, Colorimetric Test for, 592
————, in Animal Fluids, the Estimation of, Method for, 548
Acetyl Chloride and Acetone, the Action of, on the Mixed Chlorides, Separation and Estimation of Barium in Presence of Calcium and Magnesium by, 426
Acid, Acetic, Small Quantities of Acetic Anhydride in, Detection and Estimation of, 417
——, and Saponification Values in Wax, Estimation of, Simplified Process for, 355
——, Arsenic, in Presence of Arsenious Acid, Detection and Estimation of, by Means of Magnesia Mixture, 30
——, Benzoic, Estimation of, 584
——, ————, in Flesh and Fats, Detection of, 12
——, ————, in Foods, etc., Detection of, 584
——, Boric, Decomposition of Silicates by Fusion with, as Applied to Qualitative Analysis, 371
—— Citrate in Certain Organic Liquids, the Estimation of Quinine as the, 396
——, Citric, in Milk, Estimation of, 105
——, Dihydroxystearic, in Good and Poor Soils, 504
——, Diphenylamine-Sulphuric, Test, Estimation of Nitrates in Milk by Means of the, 587
——, Dimethylaminobenzene-azo-benzene Sulphonic, Use of, as a Reagent, in Microchemistry 232
——, Formic, and Methyl and Ethyl Alcohols, Detection and Estimation of, 507
——, ————, Chemical Differentiation between Fermentation Vinegars and "Vinegar Essence," Particularly with Respect to the Amount of, 455
——, ————, Estimation of, 355
——, ————, in Foods, Estimation of, 103, 496
——, Free, and Basic Alumina, in Aluminium Salts, Estimation of, 236
——, Glycuronic, Small Quantities of, Detection of, 596
——, Hippuric, as a Cause of the Failure of the Spectroscopic Test for Hæmoglobin in Urine, 417
——, Hydrated Silicic, in Clay, Determination of, 85
——, Hydrochloric, Chlorine in, Electrolytic Determination of, with the Use of a Silver Anode and Mercury Cathode, 33
——, Hydrofluoric, Preservation of Fruit Juices with, 218
——, Hypophosphorous, Estimation of Copper by Means of, 302
——, Iodic, Detection and Estimation of Thorium by Means of. (Separation of Thorium from Scandium), 436
——, Lactic, Analysis of, 556
——, ————, the Anhydride of, 235
——, Malic, the Determination of, Polarimetric Method for, and its Application in Cane and Maple Products, 498

Acid, Menthylglycuronic, Preparation of, 505
——, Nitric, and Nitrous Acid, in Water, Detection and Estimation of, 433
——, ——, Decomposition of the Bromide by, Estimation of Rubber as Tetrabromide, 361
——, ——, in Nitrocellulose, Nitroglycerol, and Other Compounds, Volumetric Determination of, 34
——, ——, in Vegetable Products, Quantitative Estimation of, 163
——, Nitrous, and Nitric Acid, in Water, Detection and Estimation of, 433
——, Oleic, Separation of, from Stearic and Palmitic Acids, 23
——, Pertitanic, the Influence of, on the Estimation of Iron in Minerals, 304
——, Phosphoric, as Magnesium-Ammonium Phosphate, Estimation of, 434
——, ————, Colorimetric Estimation of, 525
——, ————, Colorimetric Method of Estimating, 307
——, ————, Estimation of, by Neumann's Method, 525
——, ————, in Milk, Distribution of, 345
——, ————, Loss of, on Incineration in Presence of Organic Matter, Causes of, 360
——, ————, Magnesia Method for Estimation of, Criticism of, 176
——, ————, Reverted, the Estimation of, Improved, Hollemann's Method for, 605
——, ————, Soluble in 2 per Cent. Citric Acid Solution, Volumetric Estimation of, 308
——, ————, Volumetric Estimation of, 472
——, Phosphotungstic, Precipitation of Proteins, 461
——, Racemic, as a Reagent (Precipitant of Calcium and Strontium), 365
——, Salicylic, a Test for, Kobert's Reagent as, 540
——, ————, Action of, on Metallic Acids, 567
——, ————, and Phenol, Sensitive Test for, 295
——, ————, Detection of, 463
——, ————, in Fruit Juices, Estimation of, 411
——, Silicotungstic, Estimation of Aconitine by, 142
——, Sulphuric, and Sulphates, New Method of Estimating, 178
——, ————, and Zinc, Separation of, 607
——, Sulphurous, Complex Compounds of, Sulphurous Acid, and Sulphites, the Antiseptic Properties of, Experiments on, 285
——, ————, in Glucose Syrup, Estimation of, 543
——, ————, in White Wines, 351
——, Tartaric, Estimation of, 148
——, ————, in Apples, Pears, Cider, and Perry, Estimation of, 545
——, ————, Total, Estimation of, 148
——, ————, ——, ————, by Goldenberg's Method, 81
——, Telluric, Alkalimetric Estimation of, and Gravimetric Estimation of Tellurium, 248
——, Vanadic, Precipitation of, as Silver Vanadate, and Estimation of Phosphoric and Vanadic Acids in Presence of Each Other, 35
Acidity in Hydrogen Peroxide Solutions, Estimation of, 82
——— of Fats and Oils, Particularly Lubricating Oils, Estimation of, 290
——— of Tan Liquors, the Estimation of, Electrometric Method for, 121; Part II., 515
Acids, Amino-, and Peptones or Peptides, Differentiation between, 593
——, Fatty, Free, Estimation of, in the Presence of Soaps of the Alkalis and Alkaline Earths, 595
——, ——, in Soaps, Estimation of, 355
——, ——, Liquid, Separation of Solid from, 22, 114
——, ——, Soluble and Volatile, Loss of, during the Ageing of Butter, 64
——, ——, Total, in Fat, Oils, and Soaps, Estimation of, 115
——, ——, ——, in Soap, 20
——, Free Fatty, in Fats, Estimation of, 355

Acids, in Sugar-Cane Juice, Determination of, 594
——, Metallic, Action of Salicylic Acid on, 567
——, Nitric and Nitrous, the Diphenylamine Test for, Modification of, 370
——, Palmitic and Stearic, Separation of Oleic Acid from, 23
——, Phosphoric and Vanadic, in Presence of Each Other, Estimation of, and Precipitation of Vanadic Acid as Silver Vanadate, 35
—— Suitable for Use as Standards in Acidimetry, 25
 the Fatty, Effect of the Glycerol Method of Saponification upon, 425
 Total Fatty, in Soap, Estimation of, 20
 Volatile Fatty, in Fæces, Quantitative Estimation of, 505
 ———, in Wine, Estimation of, 502
 ———, of Honey, 277
Aconite, Assay of, 450
Aconitine, Estimation of, by Silicotungstic Acid, 142
Adrenaline and Allied Bases, Colour Reactions of, 18
Adulterant of African Sandal-Wood Oil, 146
Adulteration of Butter, Detection of, by Ewer's Method, 404
———— of Linseed Cakes and Linseed Meals in Belgium, 218
———— of Pepper Corns, 349
————, Turpentine, Detection of, 413
Affinity Values, Indicators for Determining, Use of, 569
Africa, German East, and Togo, Leguminous Plants Grown in, Composition of the Seeds of, 345
African Sandal-Wood Oil, Adulterant of, 146
Age of Blood-Stains, Estimation of, 455
Aged Whisky, the Substitution of Spirit for, Detection of, 546
Ageing of Butter, Loss of Soluble and Volatile Fatty Acids during, 64
Agitation, Rapid Electro-Analysis with, by Means of a Current of Gas, 600
Air, Oxygen in, Volumetric Estimation of, 569
—, Ozonised, a Constant Current of, Laboratory Apparatus for the Production of, 479
Albumin, in Urine, etc., Heller's Ring Test for the Detection of, Modification of, 152
——————, the Estimation of, Rapid Gravimetric Method for, 551
Albumins, Egg Serum, and Milk, Coagulating-Points of, and of Serum Globulin, in Ammonium Sulphate Solutions, 416
Alcohol, Ether and Benzene in, Estimation of, 19
——— -Ether, in Mineral Lubricating Oils, Proportion of Asphalt Insoluble in, 418
———, Ethyl, in the Presence of Methyl-Alcohol, Detection of, 18
———, in the Presence of Acetone, Colorimetric Test for, 592
———, Methyl, Detection of Ethyl Alcohol in the Presence of, 18
———, Propyl, Use of. Estimation of the Saponification Value of Oils, Fats, and Waxes, 290
——— -Soluble Constituents of Litmus, 25
Alcoholic Solutions, Iodine in, Estimation of, 235
Alcohols, Ethyl and Methyl, and Formic Acid, Detection and Estimation of, 507
———, Higher, Occurring in Jamaica Rum, 411
Aldehyde, *d*- Perilla, and Myrtenal, the Occurrence of, in Nature: Essential Oil of "False Camphor-Wood," 289
———— Titration, the Degree of Accuracy with which Proteins can be Estimated in Milk by, 9
Aldehydes, Aromatic, Colour Reactions of, and their Applicability in the Analysis of Spirits, 273
Algæ, Marine, and Products Derived from them, Relative Proportions of Arsenic in, 218
Aliphatic Amino Groups, Quantitative Estimation of, and its Application in the Chemistry of the Proteins, Urine, and Enzymes, 17

Alkali Carbonates, in Presence of Alkali Hydroxides or Bicarbonates, Titration of, 362
——— ———, the Use of, in Cheese Manufacture, Detection of, 218
———, Iodine, and Silver, Volumetric Solutions of, Use of Sulphur Dioxide in Checking Strengths of, 248
——— -Metal Cyanides, Sulphides in, Estimation of, 436
——— Metals, Estimation of, Elimination of Ammonium Salts Prior to, 30
———, "Titratable," in Blood, the Estimation of, New Indicator for, 415
Alkalimetric Estimation of Telluric Acid, and Gravimetric Estimation of Tellurium, 248
Alkalimetry, Standard in, Sodium Thiosulphate as, 435
Alkaline Earths and Alkalis, Soaps of the, Estimation of Free Fatty Acids in the Presence of, 595
——— Potassium Iodide Solution, Estimation of Ozone by Means of, 525
Alkaloids in Belladonna Extracts, Titration of. Estimation of Atropine, 142
Allelotropic Mixtures, the Quantitative Estimation of Enols in, Colorimetric Method for, 554
Alloys, Aluminium and Aluminium, Analysis of, 425
———, Antimony in, Volumetric Method for, 299
——— Containing Antimony and Tin, Lead in, Estimation of, 367
———, Ferrotitanium, Containing Much Silicon, Analysis of, 373
———, Iron, Estimation of Silicon in Vanadium and Molybdenum, and in their, 311
———, Other, and German Silver, the Separation of Nickel and Zinc in, Method for, 524
———, Silicon, Dissolving, 178
Almond and Apricot Kernel Oils, Note on, 263
Alumina, Basic, and Free Acid in Aluminium Salts, Estimation of, 236
———, Separation of, from Ferric Oxide, 82
Aluminium Alloys, and Aluminium, Analysis of, 425
———, Amalgamated, in Chemical Analysis, Some Uses of, 516
——— and Chromium in Ferro-Vanadium, Estimation of, 123
———, Chromium, and Iron, Precipitation of, by Ammonium Nitrite, 559
——— Hydroxide in Granular Form, Precipitation of, 299
———, Microchemical Test for, by Means of Caesium Chloride, 362
——— Salts, Free Acid and Basic Alumina in, Estimation of, 236
———, Separation of, from Beryllium, 426
———, the Separation of Iron from, and New Test for Ferrous Oxide, 373
("Amang"), Rare Earth-Containing Sands, from Malay States, 523
Amino-Acids and Peptones or Peptides, Differentiation between, 593
——— Compounds and Urine, Amino Groups in, Estimation of, and a Method for the Analysis of Proteins, 457
——— Groups, Aliphatic, Quantitative Estimation of, and its Application in the Chemistry of the Proteins, Urine, and Enzymes, 17
Ammonia and Pyridine, Separation and Estimation of, 508
——— Method, Estimation of Formaldehyde by the, 421
Ammoniacal Solution of its Oxalate, Estimation of Silver by Electrolysis of an, 308
——— Solutions, Precipitation of Zinc, Manganese, Cobalt, Nickel, Copper, and Cadmium from, by Means of Sodium Carbonate and Trimethylphenylammonium Carbonate, 599
Ammonium Nitrite, Precipitation of Aluminium, Chromium, and Iron by, 559
——— Salts, Elimination of, Prior to Estimation of Alkali Metals, 30
——— Sulphate, Estimation of Sulphur in Coal-Gas and of, 309
——— ——— Solutions, Coagulating-Points of Egg Serum and Milk Albumins, and of Serum Globulin, in, 416
Amylolytic Enzymes in Fæces, Detection of, 74

Anasthesia, Chloroform, Composition of the Gases of the Blood in, 154
Anæsthetic and Analytical Purposes, Examination of Ether for, 407
Anæsthetics, Certain Other, and Cocaine, Tests for, 2
————, Chemistry of. III. Nitrous Oxide, 503
Analysis, Capillary, 251
———, Chemical, New Method of, 376
———, Electro, 565
———, Elementary, 516
———, Qualitative, without the Use of Hydrogen Sulphide, 517
———, Toxicological, Method of Destroying Organic Matter by Means of Bromine, Particularly Applicable to, 592
Analytical Constants of Shellac, Lac Resin, and Lac-Wax, 119
———— Methods for the Estimation of Caoutchouc, Critical Investigation of, 158
Anhydride, Acetic, in Acetic Acid, Small Quantities of, Detection and Estimation of, 417
————, the, of Lactic Acid, 235
————, Reaction, an Inner, of Proteins, 514
Anhydrides, Aromatic Inner, Reaction of, Depending on their Influence on the Crystalline Form of Iodoform, 458
Anhydrous Chlorides, the Preparation of, General Method for, and its Application in Chemical Analysis, 31
Animal and Vegetable Substances, Ash in, Estimation of, 237
——— Fluids, Acetone in, Method for the Estimation of, 548
——— Organs or Plants, the Ash of, Estimation of Small Quantities of Manganese, Especially in, 369
——— Organs, Oxidising Substances in, Colorimetric Detection of, 152
——— Sizing in Paper, Detection of, Differentiation of Gelatin and Casein, 290
——— Tissues, Indophenol Oxydase of, Estimation of, 550
——— ———, Inorganic Phosphorus in, Estimation of, 284
Annual Address of the President, 43
Anode, Silver, and Mercury Cathode, Electrolytic Determination of Chlorine in Hydrochloric Acid with the Use of, 33
——, Stationary and Gauze Cathode, Rapid Determination of Nickel and Cobalt by Means of, 307
——, ————————, ———————— Silver and Cadmium by Means of, 473
——, ————, —— Mercury Cathode, Rapid Determinations and Separations by Means of, 83
Anthracite, Calorific Value of, Accurate Technical Estimation of, 518
————, Practical Value of Calorimetry in Testing, 562
Antimony, a Solid Hydride of, Attempts to Prepare, 300
———— and Arsenic Compounds, Germicidal Action of, on *Bacillus Typhosus*, 282
in Copper, Estimation of, 171
– and Tin, Alloys Containing, Estimation of Lead in, 367
– in Alloys, Volumetric Method for, 299
–, Small Quantities of, the Detection and Estimation of, Note on, 101
Antiseptic Properties of Hops, Valuation of, 65
———————— Sulphurous Acid, Sulphites, and Complex Compounds of Sulphurous Acid, Experiments on, 285
Apparatus, a New Extraction, 529
APPARATUS, ABSTRACTS, 36, 86, 126, 180, 249, 312, 376, 437, 477, 528, 568, 607
Apparatus and Methods for Sampling and Analysis of Furnace Gases, 530
————, Extraction, 609

Apparatus, Extraction, and Condenser, 378
——— for Electrolytic Estimations with Mercury Cathode, 529
———— Exact Gas Analysis, 379
———— Filtering Hygroscopic Substances or those Affected by Air, 87
———— Flue Gas Analysis, 440
———— Measuring Radioactivity, 380
———— Precipitating, Filtering, and Drying in an Inert Gas, 313
———— Rapid Electro-Analytical Determination of Metals, 181
———— Storing and Measuring Poisonous, Hygroscopic, or Volatile Liquids, 88
———— the Estimation of the Hexabromide Value of Oils, 607
—————— Melting-Point of Fats, 569
———— the Maintenance of Constant Pressures Above and Below the Atmospheric Pressure, Application to Fractional Distillation, 441
———, Laboratory, for the Production of a Constant Current of Ozonised Air, 479
———, Microdistillation, 37
———, Sand's, Electrolytic Estimation of Copper and Zinc, Using, 182
———, Vacuum Fractionation, Electrically-Heated, 253
Apples, Pears, Cider, and Perry, Tartaric Acid in, Estimation of, 545
Apricot Kernel and Almond Oils, Note on, 263
—— Kernels, Sweet, Composition of, 450
Aqueous Liquids, Automatic Extraction of, by Means of Specifically Lighter Solvents, 87
Aromatic Inner Anhydrides, Reaction of, Depending on their Influence on the Crystalline Form of Iodoform, 458
Arsacetin and Atoxyl, the Estimation of Arsenic in, Simplified Method for, 273
Arsenate, Lead, in the Products of Vines Treated with Arsenical Washes, Proportion of, 301
Arsenic, Absorption of, by Sugar Beets, 549
—— Acid, in Presence of Arsenious Acid, Detection and Estimation of, by Means of Magnesia Mixture, 30
—— and Antimony Compounds, Germicidal Action of, on *Bacillus Typhosus*, 282
———— in Copper, Estimation of, 171
—— and Iron Salts in Mineral Waters, Estimation of, 466
—— in Insecticides, Estimation of, 237
—— in Marine Algæ, and Products Derived from them, Relative Proportions of, 218
——, in Pyrites, Estimation of, 362
——, in Schweinfurt Green, Estimation of, 363
——, the Solid Hydride of, Detection of, 300
——, Vanadium in Presence of, Estimation of, and Analysis of Ferro-Vanadium, 437
Arsenical Washes, Vines Treated with, Proportion of Lead Arsenate in the Products of, 301
Arsenious Acid, Arsenic Acid in Presence of Detection and Estimation of, by Means of Magnesia Mixture, 30
Ash, in Sugar and Syrups, Estimation of, by the Determination of Electrical Conductivity, Estimation of, 298
——, in Vegetable and Animal Substances, Estimation of, 237
—— of Plants or of Animal Organs, Estimation of Small Quantities of Manganese, Especially in, 369
—— Tree, Oil from Seeds of the, 357
Asphalt Insoluble in Alcohol-Ether in Mineral Lubricating Oils, Proportion of, 418
——, Petroleum, Technical Analysis of, 294
Asphaltum, Examination of, 225

Asphaltum from Mexican Petroleum, 353
Association of Official Agricultural Chemists, the Proceedings of, Extracts from, 186
Atmosphere, the Degree of Vitiation of, Method of Measuring, 363
Atmospheric Pressure, the Maintenance of Constant Pressures Above and Below, Apparatus for, Application to Fractional Distillation, 441
Atomic Weights, International, 1911, 1
Atoxyl and Arsacetin, the Estimation of Arsenic in, Simplified Method for, 273
Atropine, Estimation of. Titration of the Alkaloids in Belladonna Extracts, 142
Automatic Extraction of Aqueous Liquids by Means of Specifically Lighter Solvents, 87
——— Inflammable Gas and Vapour Detector, New Type of, 528

Bacilli, Typhoid, in River Water, Vitality of, with Special Reference to the Question of Storage, 185
Bacillus, the Bulgarian, Note on, 221
——— *Typhosus*, Germicidal Action of Arsenic and Antimony Compounds on, 282
"Backa-Margarine," the Manufacture of, the Poisonous "Cardamon" (Maratti) Fat Employed in, Nature of, 542
Bacteriological Examination of Honey, 505
——— Investigation of Soil, 219
——— Method of Estimating Available Organic Nitrogen, 219
——— Study of Honey, 353
BACTERIOLOGICAL, PHYSIOLOGICAL, ETC., ABSTRACTS, 16, 69, 109, 152, 218, 282, 351, 415, 455, 503 548, 590
Bacteriology of Water: Its Present Position, 285
Baking-Powder and Self-Raising Flour, the Presence of Calcium Sulphate in, Report on, 255
Balsams and Essential Oils, the Examination of, Use of Miscibility Curves in, 556
Bananas, Ripe and Unripe, Composition of, 274, 541
Bang's Sugar Titration Method, the Copper Solution Used in, Preparation of, 501
Barium, in Presence of Calcium and Magnesium, Separation and Estimation of, by the Action of Acetyl Chloride and Acetone on the Mixed Chlorides, 426
———, Strontium, and Calcium, Quantitative Separation of, 364
——— Sulphate, Precipitates of, Phenomenon of Occlusion in, and its Relation to the Exact Determination of Sulphate, 426
Bark, Seychelles Cinnamon, 217
Barley, Germinating, Hordenine in, Estimation of, 162
Baroda, India, Lac Resin from, 171
Bases, Pyridine, Nicotine in the Presence of, Estimation of, 452
Basic Alumina and Free Acid in Aluminium Salts, Estimation of, 236
——— Constituents, Soluble, of the Mushroom 347
——— Slag, Phosphoric Oxide in, Citrate-Solubility of, 32
Bath, Constant-Temperature, for Use at Temperatures both Above and Below that of the Room, 249
Baths, Creosote (Dips), and Creosote Oil, the Dimethyl Sulphate Test for, 585
Bean Oil, Soya, 358
Beef and Yeast Extracts of Known Origin, Comparison of, 64
———, Australian, on a Parasite Condition (Onchocerciasis) Met with in, 129
Beer, Carbon Dioxide in, Solubility of, 404
———, Sulphites in, the Chemical Behaviour and Preservative Action of, 495
Beeswax, Analysis of, with Special Reference to East Indian and the So-Called China Beeswax, 554
———, Paraffin, Spermaceti, etc., Fat in, Detection of, 496

Beet Sugar Factory Products, Determination of Sucrose in, by Clerget's Process, Using Invertase as Hydrolyst, 120
Beets, Sugar, Absorption of Arsenic by, 549
Belgium, Linseed Cakes and Linseed Meals in, Adulteration of, 218
Belladonna Extracts, the Alkaloids in, Titration of, Estimation of Atropine, 142
Benzene and Ether, in Alcohol, Estimation of, 19
——— Derivatives, the Estimation of Halogens in, Use of Metallic Potassium in, 287
Benzidine, the Quinonoid Imonium Salts of, Estimation of the Activity of Oxidising Agents by Means of, 233
Benzoic Acid, Estimation of, 584
————, in Flesh and Fats, Detection of, 12
———— Foods, etc., 584
Berries, Coffee, the Fatty Oil and Wax Obtained from, 106
Beryllium, Separation of Aluminium from, 426
Bicarbonates, or Alkali Hydroxides, Alkali Carbonates in Presence of, Titration of, 362
Bichromate Process for Glycerol Determination, Report on, 319
Biological Examination of Honey (Enzymes), 220
——— Method for Identifying Various Fleshes, 455
——— Reactions with Serum-Precipitin and the So-Called Erythro-Precipitin for the Detection of Blood, Comparative Values of, 590
Bismuth, Estimation of. Use of Logwood in Qualitative Analysis, 603
——— Method, Estimation of Manganese by the, 430
——— Ochres from California, 174
Salicylate, Examination of, 342
Bismuthate Method, Estimation of Manganese by, 305
Blast Furnace Gas, Dust in, Determination of, 601
Blasting in Mines, the Gases Caused by, Composition of, Report on, 321
Blaud's Pill, the Composition of, Note on, 387
Bleached Flour, Digestibility of, 64
Bleaching of Flour, and the Addition of So-Called "Improvers" to Flour, Report on, 254
———, the Chemical Changes Produced in Flour by, Report on, 254
Blood, Chlorine in, Estimation of, 72
——, Detection of, 549
——, ————, by Means of Leuco-Malachite Green, 284
——, in Chloroform Anæsthesia, Condition of the Gases of, 154
——, Reagents, Influence of Metals in Presence of Hydrogen Peroxide on, 352
—— -Stains, the Age of, Estimation of, 455
——, Sugar in, Estimation of, 73
——, the Detection of, Comparative Values of the Biological Reactions with Serum Precipitin and the So-Called Erythro-Precipitin, 590
——, the Estimation of "Titratable" Alkali in, New Indicator for, 415
——, the Quantity of Calcium Contained in, Small Variations in, Method for the Estimation of, 220
Bog-Iron Ores, Analysis of, 123
Boltwood Vacuum Pump, Modified, 128
Bomb, Calorimetric, Estimation of Carbon in Steel by Means of, 562
Bombax Malabricum, Gum from, 356
Books, New, 42, 93, 326, 612
Books, Reviews of:
A Course of Practical Work in Agricultural Chemistry for Senior Students, 324

BOOKS, REVIEWS OF—*continued.*
  A Primer on Explosives for Coal-Miners, 380
  A Systematic Handbook of Volumetric Analysis, 484
  A Textbook of Botany and Pharmacognosy, 130
  Allen's Commercial Organic Analysis, Fourth Edition, Vol. III., 187, 257; Vol. IV., 380
  An Introduction to the Chemistry of Colloids, 485
  Analytical Chemistry, 611
  Arsenical Gas-Poisoning, 321
  Bacteriological and Enzyme Chemistry, 260
  Collective Index of the Journal of the Institute of Brewing, 1887-1910, 576
  Edible Fats and Oils: Their Composition, Manufacture, and Analysis, 487
  Fibres Used in Textile and Allied Industries, 322
  Huiles Minérales (Pétroles, Benzols, Brais, Paraffines, Vaselines, Ozokérite), 575
  Identification of the Commercial Dyestuffs, 385
  Laboratory Text-Book for Brewers, 441
  Là Chimica delle Sostanze Alimentari, 40
  Modern Industrial Chemistry, 325
  ——— Methods of Water Purification, 261
  National Physical Laboratory, 1910-1911, 443
  Organic Analysis Chart, 40
  Physico-Chemical Tables, Vol. II., 192
  Qualitative Chemical Analysis from the Standpoint of Solubilities, Ionisation, and Mass Action, 259
  Rubber, 443, 532
  Technical Mycology, 92
  The Chemistry and Testing of Cement, 258
  ————— of Enzymes (Allgemeine Chemie der Enzyme), 534
  The Extra Pharmacopœia, 38
  The Microscopical Examination of Food and Drugs, 384
  The Principal Starches Used as Food, 488
  The Sale of Food and Drugs Acts, 325
  The Soil Solution: The Nutrient Medium for Plant Growth, 574
  The Spark Spectra of the Metals, 41
Borax Bead Tests for Nickel and Cobalt in Admixture, Efficiency of, 173
Bordeaux Mixtures, Fungicidal Action of, 351
Boric Acid, Decomposition of Silicates by Fusion with, as Applied to Qualitative Analysis, 371
Bougies, Earthenware Filtering, Testing the Porosity of, 16
Bran, Rye and Wheat, Customs Examination of, 350
Brandy, Fusel Oil in, Detection of, 216
Bread Made from Different Varieties of Flour, the Nutritive Value of, Report to the Local Government Board on, 479
Brewers' Grains, Starch in, Estimation of, 500
British Wines, the Composition of, Note on, 60
Bromide, Decomposition of, by Nitric Acid. Estimation of Rubber as Tetrabromide, 361
Bromination Methods for the Estimation of Caoutchouc in Vulcanised Products, the Application of, 558
Bromine, Carbon, Hydrogen, Oxygen, Nitrogen, Sulphur, Chlorine, and Iodine, Analysis of a Substance Containing, 553
——— in Presence of Chlorides and Iodides, Estimation of, 241
———, ———————————, Very Small Quantities of, Estimation of, 365

Bromine, Method of Destroying Organic Matter by Means of, Particularly Applicable to Toxicological Analysis, 592
———, New Reaction of, 427
——— Value of Fats and Oils, Estimation of. Primary and Secondary Bromine Values, 19
Buchner Funnel, Addition to the, 438
Bulb, Potash, 86
Bulgarian Bacillus, Note on the, 221
Bumping, Prevention of, 608
Burette, Device for Accurately Reading a, 312
Burned Pyrites and Pyrites, Copper in, Estimation of, 366
Butter, Adulteration of, Detection of, by Ewer's Method, 404
——— and Cocoanut Oil in Butter and Margarine, Some Methods of Estimating, Observations on, 333
———, Cocoanut Oil in, Detection of, by Means of Ewer's Method, 451
———, —————, —————, ————— Fendler's Method, 451
Butter-Fat, the Composition of, the Effect of Feeding with Cocoanut Cake and Linseed Cake on, 445
———, the Ageing of, Loss of Soluble and Volatile Fatty Acids during, 64
Butters and Fats, Vegetable, Toxic Substances in, 65

Cadmium and Silver, Rapid Determination of, by Means of the Gauze Cathode and Stationary Anode, 473
———, Zinc, Manganese, Cobalt, Nickel, and Copper, Precipitation of, from Ammoniacal Solutions, by Means of Sodium Carbonate and Trimethyphenylammonium Carbonate, 599
Caesium Chloride, Microchemical Test for Aluminium by Means of, 362
Caffeine and Theobromine, Estimation of, 501
——————————, in the Presence of Each Other, Estimation of, 297
Cake, Cocoanut, and Linseed Cake, the Effect of Feeding with, on the Composition of Butter-Fat, 445
Cakes, Linseed, and Linseed Meals, in Belgium, Adulteration of, 218
——, Rapeseed, Green Colour of, 411
Calcium and Magnesium, Separation and Estimation of Barium in Presence of, by the Action of Acetyl Chloride and Acetone on the Mixed Chlorides, 426
(————— Strontium, Precipitant of), Racemic Acid as a Reagent, 365
——————————-, Sulphite Method for Separating and Identifying, 301
———, Barium, and Strontium, Quantitative Separation of, 364
——— Carbide, Use of, for Determining Moisture, 112
——— Contained in Blood, Small Variations in the Quantity of, Method for the Estimation of, 220
——— Cyanamide, Analysis of, 429, 560
——————————-, and of Norwegian Nitre, 561
——————————, Commercial, Analysis of, 76
——————————, in the Presence of Other Fertilisers, Detection and Estimation of, 466
——— Hypophosphite, Analysis of, 605
——— Nitrate and Nitrite, Commercial, Nitrate and Nitrite Nitrogen in, Estimation of, 524
———, Separation of Strontium from, 247
——— Sulphate in Baking-Powder and Self-Raising Flour, the Presence of, Report on, 258
——— Tartrate and Potassium Bitartrate, Separate Estimation of, 234
——— Very Small Amounts of, Estimation of, by Means of Permanganate, 174

California, Bismuth Ochres from, 174
Calorific Value of Anthracite, Accurate Technical Estimation of, 518
Calorimeter, Recording Thermo-Electric, 312
———, Still Water Gas, 251
Calorimetric Bomb, Estimation of Carbon in Steel by Means of, 562
Calorimetry, Practical Value of, in Testing Anthracite, 562
Camphor, Estimation of, 593
———, in Smokeless Powder, Estimation of, 420
———, Testing of, 343
"Camphor-Wood, False," Essential Oil of: The Occurrence of Myrtenal and *d*-Perilla Aldehyde in Nature, 289
Candelilla Wax, 598
Candle Turbidimeter, Jackson's, Determination of Sulphur in Coal by Means of, 519
Cane and Maple Products, Polarimetric Method for the Determination of Malic Acid, and its Application in, 498
Canned Foods, Tin in, Estimation of, 149
Cantharides and Tinctures, Quantitative Estimation of Cantharidine in, 343
———, Tincture and Oil of, Estimation of Cantharidine in, 404
Caoutchouc as Tetrabromide, Direct Estimation of, 224
———, ———, Estimation of, 159, 509
———, Crude, Analysis of, 159
———, in Vulcanised Products, the Estimation of, the Application of Bromination Methods for, 558
———, in Vulcanised Rubber Articles, Direct Estimation of, 113
———, in Vulcanised Rubber Goods, Direct Estimation of, 75
———, Nitrosite and its Analytical Application, 287
———, Tetrabromide, Hubener's, Notes on, 235
———, the Analytical Methods for the Estimation of, Critical Investigation of, 158
Cap-Compositions, Fulminate, Analysis of, 595
Capillary Analysis, 251
Caramel and Prune Juice in Vanilla Flavouring Extracts, Detection of, 281
———, Preparation and Constitution of, and its Estimation in Sugar Juices and Syrups, 273
Carbalkyloxy Derivatives, Use of, for the Quantitative Estimation of Hydroxyl Groups, 227
Carbinol, Phenyl-ethyl, Estimation of Morphine by Extraction with, 587
Carbon and Nitrogen in Organic Compounds, Estimation of, 593
——— Sulphur in High-Grade Alloys of Tungsten, Molybdenum, and Vanadium, with Iron, Estimation of, 31
——— Dioxide in Beer, Solubility of, 404
——— Dioxide in Carbonates and Nitrogen Pentoxide in Nitrates, the Estimation of, by Loss on Ignition, Application of Sodium Paratungstate in, 526
——— Dioxide Recorder, Multiple, 252
———, Hydrogen, Oxygen, and Nitrogen, Organic Substances Containing, New Method of Elementary Analysis of, 553
———, Hydrogen, Oxygen, Nitrogen, and Sulphur, Analysis of a Substance Containing, 553
———, Hydrogen, Oxygen, Nitrogen, Sulphur, Chlorine, Bromine, and Iodine, Analysis of a Substance Containing, 553
———, in Organic Substances, the Estimation of, Wet Method for, 354
———, in Steel, Estimation of, by Means of a Calorimetric Bomb, 562
———, Microscopical Examination and Identification of, 428
——— Monoxide Detector, 608

Carbon Monoxide Hæmoglobin, and Oxyhæmoglobin, the Spectroscopic Differentiation between, and Hæmochromogen, 550

———, Oxygen, Hydrogen, and Nitrogen, Organic Substances Containing, New Method of Elementary Analysis of, 553

Carbonates, Alkali, in Presence of Alkali Hydroxides or Bicarbonates, Titration of, 362

————, ———, the Use of, in Cheese Manufacture, Detection of, 218

————, Carbon Dioxide in, and Nitrogen Pentoxide in Nitrates, the Estimation of, by Loss on Ignition, Application of Sodium Paratungstate in, 526

Carbonylferrocyanides: Their Extraction and Analysis, 157

Carborundum, in Coke Crucible Scrap, Estimation of, 31

"Cardamon" (Maratti) Fat, Poisonous, Employed in the Manufacture of "Backa Margarine," Nature of, 542

Carius, the Method of, Estimation of Sulphur by, 296

Casein and Gelatin, Differentiation of. Detection of Animal Sizing in Paper, 290

——— Product, 406

———, Quantitative Estimation of Tannin by Means of, 123

Cassia Fistula, the Pulp of, Composition of, 213

Catalase in Milk, Estimation of, 284

Cathode, Gauze, and Stationary Anode, Rapid Determination of Nickel and Cobalt by Means of, 307

———, ———, ——————, Rapid Determination of Silver and Cadmium by Means of, 473

———, Mercury, and Silver Anode, Electrolytic Determination of Chlorine in Hydrochloric Acid with the Use of, 33

———, ————, and Stationary Anode, Rapid Determinations and Separations by Means of, 83

———, ————, Electrolytic Estimations with, Apparatus for, 529

Caulophyllin, a Reaction for, 270

Ceara Rubber, 424

Cellulose, Estimation of, by Simon and Lohrisch's Method, 77

Cement, Ferric Oxide in, Rapid Estimation of, 563

———, Portland, Uniform Methods of Analysis for, 247

Cereals and Wood-Fungi, Pentosans and Methylpentosans in, Estimation of, 460

Cerium, Separation of, by Potassium Permanganate, 429

———, —————, New Method for, 518

Changes which Mercury Compounds undergo when in Contact with Organic Matters, 154

Cheese, Gorgonzola, Note on, 61

——— Manufacture, the Use of Alkali Carbonates in, Detection of, 218

———, Roquefort, Composition of, 542

Chemical Analysis, Some Uses of Amalgamated Aluminium in, 516

———— Changes Produced in Flour by Bleaching, Report on, 254

———— Disinfectants, Study of, Contribution to, 69

——————, ———, —————, Second Paper, 283

Chicken and Turkey Fats, Constants of, 213

China, the So-Called, and East Indian Beeswax, Analysis of Beeswax, with Special Reference to, 554

Chlorate, Potassium, Estimation of Potassium Perchlorate in, 248

Chlorates, Chlorides, and Perchlorates, in the Presence of Each Other, Estimation of, 365

Chloride, Acetyl, and Acetone, the Action of, on the Mixed Chlorides, Separation and Estimation of Barium in Presence of Calcium and Magnesium by, 426

————, Caesium, Microchemical Test of Aluminium by Means of, 362

Chloride, Mercuric, Volumetric Estimation of, 34
———, Stannous Estimation of Tungsten by Means of, 310
Chlorides and Iodides, Bromine in Presence of, Estimation of, 241
——————, Very Small Quantities of Bromine in Presence of, Estimation of, 365
————, Anhydrous, the Preparation of, General Method for, and its Application in Chemical Analysis, 31
————, Chlorates, and Perchlorates, in the Presence of Each Other, Estimation of, 365
————, Mixed, the Action of Acetyl Chloride and Acetone on, Separation and Estimation of Barium in Presence of Calcium and Magnesium by, 426
Chlorine, Carbon, Hydrogen, Oxygen, Nitrogen, Sulphur, Bromine, and Iodine, Analysis of a Substance Containing, 553
———— in Blood, Estimation of, 72
———— in Hydrochloric Acid, Electrolytic Determination of, with the Use of a Silver Anode and Mercury Cathode, 33
Chloriplatinate, Potassium, Reduction of, 526
Chloroform Anæsthesia, Composition of the Gases of the Blood in, 154
Chocolate, Cocoanut Oil in, the Detection of, Note on, 275
Chromate, Lead as, Volumetric Determination of, 602
Chrome Ironstone, Chromium in, Estimation of, 428
———— Tungsten Steel, Chromium in, Estimation of, 83
Chrome-Vanadium and Vanadium Steels, Manganese in, Estimation of, 604
————————————————, Vanadium in, Determination of, 475
Chromium, Aluminium, and Iron, Precipitation of, by Ammonium Nitrite, 559
-- and Aluminium in Ferro-Vanadium, Estimation of, 123
————, in Chrome Ironstone, Estimation of, 428
————, ———— Tungsten Steel, Estimation of, 83
———— in Steel, Detection of, 428
——, Minute Quantities of, Colorimetric Estimation of, 238
Cider, Apples, Pears, and Perry, Tartaric Acid in, Estimation of, 545
—— Vinegar, 589
Cinnamon Bark, Seychelles, 217
Citrate-Solubility of Phosphoric Oxide in Basic Slag, 32
Citric Acid in Milk, Estimation of, 105
———— Solution, 2 per Cent., Phosphoric Acid Soluble in, Volumetric Estimation of, 308
Classen Dish, Holder for, 180
Clay, Hydrated Silicic Acid in, Determination of, 85
Clerget's Process, Determination of Sucrose in Beet Sugar Factory Products by, Using Invertase as Hydrolyst, 120
Coagulating-Points of Egg, Serum, and Milk Albumins, and of Serum Globulin, in Ammonium Sulphate Solutions, 416
Coal-Gas, Crude, Naphthalene in, Estimation of, 510
————, Sulphur in, Estimation of, and of Ammonium Sulphate, 309
————, Total Sulphur in, the Estimation of, "Perhydrol" for, 309
Coal, Purchase of, under Specifications, 229
——, Sampling of, in the Mine, 527
——, Sulphur in, Determination of, by Means of Jackson's Candle Turbidimeter, 519
—— -Tar Creosote, Testing of, 419
—— Tar, the Residues from the Distillation of, Estimation of Rosin in, 463
—— Tars, and Refined Tars, Oils, and Pitches Derived Therefrom, Methods for Testing, 298

Cobalt and Nickel, Gravimetric Estimation of, 598
——————— in Admixture, Borax Bead Tests for, Efficiency of, 173
———————, Rapid Determination of, by Means of the Gauze Cathode and Stationary Anode, 307
———————, the Differentiation of, New Reaction for, 238
Cobalt Reaction, Modified, for the Detection of Very Small Quantities of Hydrogen Peroxide, 598
———, Zinc, Manganese, Nickel, Copper, and Cadmium, Precipitation of, from Ammoniacal Solutions, by Means of Sodium Carbonate, and Trimethylphenylammonium Carbonate, 599
Cobalti-Nitrite, Small Quantities of Potassium as, Detection of, 606
Coca Leaves, Analysis of, 274
Cocaine and Certain Other Anæsthetics, Tests for, 2
————- Substitutes, Identification of, 405
——— Solutions, Precipitation of, with Platinum Chloride, 354
Cocoa Powder, Husk in, Estimation of, 275
—— Products, Husk and Germ in, Quantity of, 214
Cocoanut Cake and Linseed Cake, the Effect of Feeding with, on the Composition of Butter-Fat, 445
——— Oil and Butter in Butter and Margarine, Some Methods of Estimating, Observations on, 333
—————- in Butter, Detection of, by Means of Ewer's Method, 451
——————, ————-, ———— Fendler's Method, 451
————— in Chocolate, the Detection of, Note on, 275
—————- in Lard, Estimation of, by Means of the "Ethyl Ester Value," 106
—————-, Shrewsbury and Knapp's Process for the Estimation of, Examination of, 195
Codeine, in Opium, the Determination of, 489
Cod-Liver Oil, Analysis of, by Means of its Miscibility Curve, 275
Coffee Berries, the Fatty Oil, and Wax obtained from, 106
——, Glazed, Resin in, Detection of, 275
Coke Crucible Scrap, Carborundum in, Estimation of, 31
—— Furnaces, the Gases from, Cyanogen Compounds in, Estimation of, 77
Colchicine, in Toxicology, Detection of, 73
Colophony, Colour Reactions of Certain Resins with Halphen's Reagent for, 170
Colorimeter, New, 478
Colorimetric Detection of Oxidising Substances in Animal Organs, 152
—————- Estimation of Lactose in Urine and in Milk, 592
—————————— of Minute Quantities of Chromium, 238
—————————— of Phosphoric Acid, 525
—————————— of Strychnine, 144
—————————— of Sugar, Creatine, and Creatinine in Urine, 591
—————————— of Titanium, Sensitiveness of, 310
——————————————, Use of Methyl Orange in, 310
—————- Method of Estimating Phosphoric Acid, 307
———————— for Estimating Urochrome and Urochromogen, and Relation of the Normal Yellow Pigmentary Substances of Urine to the Diazo-Reaction, 223
———————— for the Estimation of the Molecular Weight of Polysaccharides, 360
———————— for Vanadium in Iron and Steel, 473
—————- Test for Alcohol, in the Presence of Acetone, 592
Colostrum, Behaviour of Schardinger's Reagent towards, 221
Colour Reaction, Characteristic, of Ovolecithin, 460

Colour Reaction of Proteins with Sodium Nitroprusside, 235
——— Reactions, Differentiation of Minerals by Means of, 242
—— of Adrenaline and Allied Bases, 18
—— of Aromatic Aldehydes and their Applicability in the Analysis of Spirits, 273
—— of Certain Resins with Halphen's Reagent for Colophony, 170
—— of Edible Fats and Oils, 406
—— of Strychnine, Influence of Certain Compounds upon the, 296
——— Scale Used with Schaeffer's Curd Tester, Control Tests of, 378
Colouring Matters, Certain, Used in Foods, Report on the Solubility, Extraction, and Reactions of, 186
Columbium and Tantalum, Indirect Method for Determining, 239
———, ——— for the Estimation of, 310
———, Separation of, 239
Combustible Gases and Vapour, Safety Lamps or Other Apparatus for the Detection of, Methods of Testing, 528
Commercial Absinthe, Analysis of, 12
——— Calcium Cyanamide, Analysis of, 76
——— Nitrate and Nitrite, Nitrate and Nitrite Nitrogen in, Estimation of, 524
——— Dextrins, Analysis of, 276
——— Gelatins, Sulphur Dioxide in, 271
——— Oxygen, Analytical Examination of, 469
——— Peptones, Certain, Physical Constants and Analyses of, 111
——— Starch Syrup, the So-Called Gallisin (Glucosin) Occurring in, Composition of, 543
Concentrate, Wolfram, the Assay of, 398
Condensed Milk, Sweetened, Analysis of, 138
——— Milks, an Inquiry as to, Report to the Local Government Board on; with Special Reference to their Use as Infants' Foods, 572
Condenser and Extraction Apparatus, 378
———, Improved Soxhlet, 528
Conductivity, Electrical, and Osmotic Pressure, Application of, in the Examination of Soils, 184
———, ———, of Soils, Determination of, 183
———, ———, the Determination of, Estimation of Ash in Sugar and Syrups by, 298
Conference, International, on Food Analysis, 536
Congress of Applied Chemistry, Eighth International, 386
Conifer Fatty Oils, Characteristics of, 511
Constant, New, in the Analysis of Pepper, 147
Constant Pressures Above and Below the Atmospheric Pressure, the Maintenance of, Apparatus for, Application to Fractional Distillation, 441
———-Temperature Bath for Use at Temperatures both Above and Below that of the Room, 249
——— Temperature Drying-Oven, New Form of, 126
Convallamarin and Convallarin: Glucoside Reactions, 289
Copper and Zinc, Electrolytic Estimation of, Using Sand's Apparatus, 181
Copper, Arsenic and Antimony in, Estimation of, 171
———, Estimation of, by Means of Hypophosphorous Acid, 302
——— in Pyrites and in Burned Pyrites, Estimation of, 366
———, Ores Containing Much, Pot Assay of Gold and Silver in, 240
———, Refined, Exact Electrolytic Assay of. I. Standard Method, 172
——— Solution Used in Bang's Sugar Titration Method, Preparation of, 501

Copper, Zinc, Manganese, Cobalt, Nickel, and Cadmium, Precipitation of, from Ammoniacal Solutions, by Means of Sodium Carbonate and Trimethyphenylammonium Carbonate, 599
Coprah, Examination of, 512
Cows' Milk, Oxydase and Reductase Occurring in, 16
———, on the Peroxydase of, and its Detection by Means of the *p*-Phenylenediamine Test, 222
Cows' Milk, the Detection of Goats' Milk in, and Composition of Goats' Milk, 218
Creatine in Pathological Urine, Direct Estimation of, 504
——— Sugar, and Creatinine, in Urine, Colorimetric Estimation of, 591
Creatinine, the Folin Method of Estimating, Sources of Error in, 288
Creosote and Tar Oils Available for Wood Preservation, Characters of, 228
——— Baths (Dips) and Creosote Oil, the Dimethyl Sulphate Test for, 585
———, Coal-Tar, Testing of, 419
——— Oil and Creosote Baths (Dips), the Dimethyl Sulphate Test for, 585
p-Cresol and Phenol in the Presence of Each Other, Estimation of, 79
Crucible, Gooch, for Micro-Analysis, 180
Crude Caoutchouc, Analysis of, 159
——— Coal-Gas, Naphthalene in, Estimation of, 510
——— Glycerol, Analysis of, Report on, 314
Cryoscopic Examination of Milk, 345
"Cupferron," in Quantitative Analysis, Application of, 175
———, Quantitative Separation by Means of, 520
Cupreïne, New Test for, 113
Curd-Tester, Schaeffer's, the Colour Scale Used with, Control Tests of, 378
Customs Examination of Rye and Wheat Bran, 350
Cyanamide, Calcium, Analysis of, 429, 560
———, ———, ———, and of Norwegian Nitre, 561
———, ———, Commercial, Analysis of, 76
———, ———, in the Presence of Other Fertilisers, Detection and Estimation of, 466
Cyanide, Hydrogen, Traces of, The Detection of, 266
———, Potassium, in Presence of Potassium Ferrocyanide, Titration of, 460
Cyanides, Alkali-Metal, Sulphides in, Estimation of, 436
Cyanogen Compounds in the Gases from Coke Furnaces, Estimation of, 77
Cylinder Oils, Dark Mineral, Estimation of Tarry Substances in, 298

Dammar Resin, Different Varieties of, Characteristics of, 422
Dangers Connected with the Storage and Transport of Ferrosilicon, 302
("Death Camas"), *Zygadenus Intermedius*, Analysis of, 156
Denitrifying Organisms, the Gases Produced by, Occurrence, Detection, and Estimation of Nitrous Oxide in, 591
Density of Liquids, Determination of, 439
Deposits Resulting from the Lubrication of Engines, 230
Detector, Automatic Inflammable Gas and Vapour, New Type of, 528
———, Carbon Monoxide, 608
Detonation, the Velocity of, Study of, 288
Device for Accurately Reading a Burette, 312
Dextrin in Foods, the Detection of, Estimation of Sucrose and, 588
Dextrins, Commercial, Analysis of, 276
Dextrose, Normal Weight of, 78
Diazo-Reaction, Relation of the Normal Yellow Pigmentary Substances of Urine to, and a Colorimetric Method for Estimating Urochrome and Urochromogen, 223
Digestibility of Bleached Flour, 64

Dihydroxystearic Acid in Good and Poor Soils, 504
Dimethyl Sulphate Test for Creosote Oil and Creosote Baths (Dips), 585
Dimethylaminobenzene-azo-benzene Sulphonic Acid, Use of, as a Reagent in Microchemistry, 232
Dinner, Anniversary, 96
Diphenylamine-Sulphuric Acid Test, Detection and Estimation of Nitrates in Milk by Means of the, 67
——————————————, Estimation of Nitrates in Milk by Means of the, 587
Diphenylamine Test for Nitrous and Nitric Acids, Modification of, 370
(Dips) Creosote Baths and Creosote Oil, the Dimethyl Sulphate Test for, 585
Disaccharides, Small Quantities of, Detection of, 594
Dish, Classen, Holder for, 180
Disinfectants, Chemical, Study of, Contribution to, 69
—————, ————, ———, ——————, Second Paper, 283
Dissolved Oxygen, Estimation of, 245
Distillation, Fractional, Application to, Apparatus for the Maintenance of Constant Pressures Above and Below the Atmospheric Pressure, 441
————— of Tar, Flask for, 529
————— of Tars Containing Water, 171
Doughing Value of Flour, Estimation of, 216
Dried Milk Powders, Composition of, 279
Drinking Water, Sterilisation of, by Means of Ultra-Violet Rays, 457
(Drop Hammer), the Impact Machine, Sensitiveness Tests by, 314
Drugs, etc., Quinine in, Volumetric Estimation of, 143
Drying-Oven, Constant Temperature, New Form of, 126
———, Precipitating, and Filtering, in an Inert Gas, Apparatus for, 313
Dust in Blast-Furnace Gas, Determination of, 601

Earth-Containing Sands, Rare, ("Amang"), from Malay States, 523
——— Minerals, Rare, the Determination and Analysis of, Use of Sulphur Monochloride in, 567
Earths, Alkaline, and Alkalis, Soaps of the, Estimation of Free Fatty Acids in the Presence of, 595
East Indian and the So-Called China Beeswax, Analysis of Beeswax, with Special Reference to, 554
Edible Fats and Oils, Colour Reactions of, 406
Effluents, Sewage, Containing Nitrites, the Amount of Dissolved Oxygen Absorbed by the Determination of, and of the Amount of Nitrite in Sewage Effluents and Water, 393
Egg, Serum, and Milk Albumins, Coagulating-Points of, and of Serum Globulin, in Ammonium Sulphate Solutions, 416
Egypt, Survey Department, 1910, the Work of the Laboratories, Report on, 532
Elastin, Detection of Active Pepsin in the Intestines by Means of, 551
———— in the Detection of Proteolytic Enzymes, Use of, 590
Electrical Conductivity and Osmotic Pressure in the Examination of Soils, Examination of, 184
—————————— of Soils, Determination of, 183
——————————, the Determination of, Estimation of Ash in Sugar and Syrups by, 298
Electrically-Heated Vacuum Fractionation Apparatus, 253
Electro Analysis, 565
———————, Rapid, under Reduced Pressure, 601
———————, ———, with Agitation by Means of a Current of Gas, 600

Electro Analysis, Rapid, with Stationary Electrodes, Adaptation of the Tap-Funnel to, 313
——— -Analytical Determination of Metals, Rapid, Apparatus for, 181
Electrolytic Assay, Exact, of Refined Copper. I. Standard Method, 172
——— Determination of Chlorine in Hydrochloric Acid with the Use of a Silver Anode and Mercury Cathode, 33
——— Estimation of Copper and Zinc, Using Sand's Apparatus, 182
——— ——— of Manganese, 177
——— ——— of Metals, Exact Method for, 370
——— Estimations with Mercury Cathode, Apparatus for, 529
——— Separations, 32
Electrometric Method for the Estimation of the Acidity of Tan Liquors, 121; Part II., 515
Elements, the Sulphides of which are Precipitated by Hydrogen Sulphides in Acid Solution, Rapid Qualitative Detection of, 568
Enamel, Analysis of, 33
Engines, the Lubrication of, Deposits Resulting from, 230
Enols in Allelotropic Mixtures, the Quantitative Estimation of, Colorimetric Method for, 554
Enzymatic Activity of Nuclease, Estimation of, 109
Enzymes, Amylolytic, in Fæces, Detection of, 74
———, Honey, Biological Examination of, 220
———, One or More, Method for Ascertaining whether a Liquid Contains, 110
———, Proteins, and Urine, the Chemistry of, Quantitative Estimation of Aliphatic Amino Groups, and its Application in, 17
-, Proteolytic, the Detection of, Use of Elastin in, 590
Errata, 141, 610
Error, Some Sources of, in Gasometric Estimation of Nitrate by Schloesing's Method, and of Nitrite by Piccini's Method, 525
———, ———, in the Folin Method of Estimating Creatinine, 288
Erythro-Precipitin, the So-Called, and Serum-Precipitin, the Biological Reactions with, for the Detection of Blood, Comparative Values of, 590
Essence, Rosin, Turpentine Oil, and Wood Turpentine Oil, Reaction of, 107
Essential Oil of "False Camphor Wood": The Occurrence of Myrtenal and *d*-Perilla Aldehyde in Nature, 289
——— of Orange Flowers, 67
——— of *Santolina Chamæcyparissus*, 589
——— of *Schinus Molle* (L.), 68
——— of Spanish Marjoram (*Thymus Mastichina*, L.), 69
——— Oils and Balsams, the Examination of, Use of Miscibility Curves in, 556
——— and Philippine Terpenes, 15
———, Estimation of, 288
, Ketones in, Estimation of, 514
Ether and Benzene in Alcohol, Estimation of, 19
——— in the Analysis of Metals, Use of, 431
———, Quantitative Analysis of Gold with the Use of, 303
———, Separation of Iron and Vanadium by Means of, 522
———, The Examination of, for Anæsthetic and Analytical Purposes, 407
———, Traces of Water in, the Accurate Estimation of, Method for, 420
Ethyl Alcohol in the Presence of Methyl Alcohol, Detection of, 18
——— and Methyl Alcohols and Formic Acid, Detection and Estimation of, 507
"Ethyl Ester Value," Estimation of Cocoanut Oil in Lard by Means of the, 106
Evaporated Milk and Milk Products, Analysis of, 278
Ewer's Method, Detection of Adulteration of Butter by, 404

Ewer's Method, Detection of Cocoanut Oil in Butter by Means of, 451
Experiments on the Precipitation of Proteins, 295
Explosives in Various Countries, the (General) Stability of, Methods of Testing, 79
Extinguishing the Flame of Petroleum Spirit, 252
Extraction Apparatus, 609
—————— and Condenser, 378
——————, New, 529
————, Automatic, of Aqueous Liquids by Means of Specifically Lighter Solvents, 87
Extracts and Powder, Liquorice, Glycyrrhizin and Sugars in, Estimation of, 409
———, Belladonna, Titration of the Alkaloids in. Estimation of Atropine, 142
——— of Known Origin, Beef and Yeast, Comparison of, 64
——————————, Meat and Yeast, Comparison of, 104
———, Tobacco, and Nicotine Solutions, Nicotine in, Estimation of, 544
———, ———, and their Value, 411
———, ———, Nicotine in, Estimation of, 348
———, ———, ————, Polarimetric Estimation of, 279
———, Vanilla Flavouring, Prune Juice and Caramel in, Detection of, 281

Factors, Surface, The Use of, and Errors in Determining Sizes of Mineral Grains, 182
Fæces, Amylolytic Enzymes in, Detection of, 74
——, Preservation of, 74
——, Volatile Fatty Acids in, Quantitative Estimation of, 505
Fat from the Seeds of *Lophira Alata* (Banks), Composition of, 493
— in Beeswax, Paraffin, Spermaceti, etc., Detection of, 496
— in Feeding Stuffs, Estimation of, by Means of Trichlorethylene, 542
— in Milk, the Estimation of, Wendler's Method for, 14
— in Pathological Urine, Estimation of, 417
— in (Maratti), Cardamon, Poisonous, Employed in the Manufacture of "Backa-Margarine," Nature of, 542
—, Oils, and Soaps, the Total Fatty Acids in, Estimation of, 115
— Pitch, Petroleum Pitch in, Detection of, 462
Fats and Butters, Vegetable, Toxic Substances in, 65
—— and Flesh, Benzoic Acid in, Detection of, 12
—— and Oils, Analysis of, 164
—————, Edible, Colour Reactions of, 406
—————, Particularly Lubricating Oils, the Acidity of, Estimation of, 290
—————, Some Indian, 108
—————, the Bromine Value of, Estimation of. Primary and Secondary Bromine Values, 19
—————, Vegetable, Some Little-Known Characteristics of, 21
Fats, Chicken and Turkey, Constants of, 213
——, Estimation of the Reichert-Meissl Value of, by Means of Glycerol-Potassium Hydroxide, 542
——, Free Fatty Acids in, Estimation of, 355
——, Melting-Point of, Apparatus for the Estimation of, 569
——, Oils, and Soaps, Train Oil in, Detection of, 359
——, Oils, and Waxes, the Saponification Value of, Estimation of. Use of Propyl Alcohol, 290
Fatty Acids, Effect of the Glycerol Method of Saponification upon the, 425
————, Free, in Fats, Estimation of, 355
————, ——, Estimation of, in the Presence of Soaps of the Alkalis and Alkaline Earths, Estimation of, 595
———— in Soaps, Estimation of, 355
————, Liquid, Separation of Solid from, 22, 114

Fatty Acids, Soluble and Volatile, Loss of, during the Ageing of Butter, 64
————, Total, in Soap, Estimation of, 20
————, ——, in Soaps, Fat, and Oils, Estimation of, 115
————, Volatile, in Fæces, Quantitative Estimation of, 505
—— Oil and Wax Obtained from Coffee Berries, 106
—— Oils, Conifer, Characteristics of, 511
————, Rosin in, Estimation of, 557
Feeding Stuffs, Fat in, Estimation of, by Means of Trichlorethylene, 542
————, Sugars in, Estimation of, 453
Fehling's Solution, Estimation of Pentosans with the Aid of, 161
Fendler's Method, Detection of Cocoanut Oil in Butter by Means of, 451
Fermentation Experiments, Quantitative, Flask for, 568
Fermentation Vinegars and "Vinegar Essence," Chemical Differentiation between, Particularly with Respect to the Amount of Formic Acid, 455
Ferric Iron, Volumetric Estimation of, by Permanganate, after Reduction with Zinc, 521
—— Oxide in Cement, Rapid Estimation of, 563
————, Separation of Alumina from, 82
—— Sulphate as Standard for Permanganate Solutions, 520
Ferrocyanide, Potassium, Potassium Cyanide in Presence of, Titration of, 460
————, Pure Potassium, Preparation of, and its Analytical Control, 564
Ferrocyanides and Thiocyanates, Simultaneous Estimation of, 563
Ferrosilicon, the Storage and Transport of, Dangers Connected with, 302
Ferrotitanium Alloys Containing Much Silicon, Analysis of, 373
Ferro-Uranium, Analysis of, 125
Ferro-Vanadium, Aluminium and Chromium in, Estimation of, 123
————, Analysis of, and Estimation of Vanadium in Presence of Arsenic, 437
Ferro-Zircon, Analysis of, 124
Ferrous Iron, in Silicate Rocks, Determination of, 602
—— Oxide in Silicates, Titration of, by the Pebal-Dölter Method, Note on, 372
————, New Test for, and the Separation of Iron from Aluminium, 373
—— Salts, Oxidation of, 467
Fertilisers, Other, Calcium Cyanamide in the Presence of, Detection and Estimation of, 466
Fiehe's Reaction, Examination of Honey by, 451
Fillers (Mineral Matter) in Rubber Mixtures, Estimation of, 558
Filtering Funnel, Rapid, 609
Filtering Hygroscopic Substances or those Affected by Air, Apparatus for, 87
————, Precipitating, and Drying in an Inert Gas, Apparatus for, 313
Filters, Earthenware (Bougies), Testing the Porosity of, 16
Finnish Turpentine, the Examination of, Note on, 577
Flame of Petroleum Spirit, Extinguishing the, 252
Flask for Distillation of Tar, 529
Flask for Quantitative Fermentation Experiments, 568
——, Measuring, for Use in Determining Iodine Values of Oils, 379
Flesh and Fats, Benzoic Acid in, Detection of, 12
Fleshes, Various, Biological Method for Identifying, 455
Flour, Bleached, Digestibility of, 64
——, Bread Made from Different Varieties of, the Nutritive Value of, Report to the Local Government Board on, 479
——, Self-Raising, and Baking-Powder, the Presence of Calcium Sulphate in, Report on, 255
——, the Bleaching of, and the Addition of So-Called "Improvers" to Flour, Report on, 254

Flour, the Chemical Changes Produced in, by Bleaching, Report on, 254
——, the Doughing Value of, Estimation of, 216
——, Wheaten, the Quality of, Method of Estimating, 543
Flowers, Orange, Essential Oil of, 67
Flue Gas Analysis, Apparatus for, 440
Fluids, Animal, Acetone in, the Estimation of, Method for, 548
Fluorine, Estimation of, 303
—— in the Human Body, Distribution of, 75
Fodder-Stuffs, Poisonous Ricinus Constituents in, Detection of, 506
Folin Method of Estimating Creatinine, Sources of Error in, 288
Food Analysis, International Conference on, 536
FOODS AND DRUGS ANALYSIS, ABSTRACTS, 12, 64, 103, 142, 214, 272, 342, 404, 450, 495, 541, 584
Foods, Canned, Tin in, Estimation of, 149
——, Certain Colouring Matters Used in, Report on the Solubility, Extraction, and Reactions of, 186
——, etc., Benzoic Acid in, Detection of, 584
——, Formic Acid in, Estimation of, 103, 496
——, Inspectors of, on the Work of, 128
——, the Detection of Dextrin in, and Estimation of Sucrose, 588
Formaldehyde, Estimation of, 114, 595
——, ——, by the Ammonia Method, 421
Formic Acid and Methyl and Ethyl Alcohols, Detection and Estimation of, 507
——, Chemical Differentiation between Fermentation Vinegars and "Vinegar Essence," Particularly with Respect to the Amount of, 455
——, Estimation of, 355
in Foods, Estimation of, 103, 496
Fractional Distillation, Application to, Apparatus for the Maintenance of Constant Pressures above and below the Atmospheric Pressure, 441
Fractionation Apparatus, Vacuum, Electrically-Heated, 233
Free Acid and Basic Alumina in Aluminium Salts, Estimation of, 236
Freezing Mixtures, 127
French Wool-Grease Oleines, Rosin Oils in, Detection of, 30
Frey's, Henry C., Method of Estimating Petroleum in Turpentine, Note on, 137
Fruit Juices. Preservation of with Hydrofluoric Acid, 218
——, Salicylic Acid in, Estimation of, 411
Fuel Oil and Natural Gas, Specifications and Sampling of, 555
Fulminate Cap-Compositions, Analysis of, 595
Fungi, Wood, and Cereals, Pentosans and Methylpentosans in, Estimation of, 460
——, Certain, Pentosans of, 162
Fungicidal Action of Bordeaux Mixtures, 351
Funnel, Filtering, Rapid, 609
——, Separatory, Modified, and Washer for Heavy Liquids, 89
——, the Buchner, Addition to, 438
Furfural Method for the Estimation of Pentosans, Modification of, 161
Furnace, Blast, Gas, Dust in, Determination of, 601
—— Gases, Sampling and Analysis of, Apparatus and Methods for, 530
Furnaces, Coke, the Gases frnm, Estimation of Cyanogen Compounds in, 77
Fusel Oil in Brandy, Detection of, 216

Gallisin (Glucosin), the So-called, Occurring in Commercial Starch Syrup, Composition of, 543
Galvanised Iron, Method for Testing, to Replace the Preece Test, 304
Gambier, Microscopical Examination of, 344

Gas, An Inert, Precipitating, Filtering, and Drying in, Apparatus for, 313
——, Analysis, Exact, Apparatus for, 379
——, ————, Flue, Apparatus for, 440
——, Blast Furnace, Dust in, Determination of, 601
——, Calorimeter, Still Water, 250
—— -Engines and Motor-Cars, Lubricating Oils for, 291
—— in Sealed Tube Reactions, Detection of, 531
—— Mixtures, Hydrogen in, Estimation of, 84
——, Natural, and Fuel Oil, Specifications and Sampling of, 555
—— -Pressure Regulators, Simple Forms of, 477
——, Rapid Electro-Analysis with Agitations by Means of a Current of, 600
—— Regulator for Thermostats, 127
——, Tar and Tar Constituents in, New Method of Estimating, 299
Gases Caused by Blasting in Mines, Composition of, Report on, 321
———, Combustible, and Vapour, Safety Lamps or Other Apparatus for the Detection of, Methods of Testing, 528
——— from Coke Furnaces, Cyanogen Compounds in, Estimation of, 77
———, Furnace, Sampling and Analysis of, Apparatus and Methods for, 530
——— of the Blood in Chloroform Anæsthesia, Composition of, 154
——— Produced by Denitrifying Organisms, Nitrous Oxide in, Occurrence, Detection, and Estimation of, 591
Gelatin and Casein, Differentiation of. Detection of Animal Sizing in Paper, 290
————, Estimation of, 585
Gelatine, Analysis of, 289
Gelatins, Analysis of, 116
————, Commercial, Sulphur Dioxide in, 271
Germ and Husk in Cocoa Products, Quantity of, 214
German Margarine, the Poisonous Properties of Certain Brands of, Cause of, 216
———— Silver and Other Alloys, the Separation of Nickel and Zinc in, Method for, 524
Germicidal Action of Arsenic and Antimony Compounds on *Bacillus Typhosus*, 282
—————- and Insecticidal Values of Different Soaps, with or without Associated Active Agents, 505
Germinating Barley, Hordenine in, Estimation of, 162
Ghee, Further Analyses of, 392
Glance, Molybdenum, Analysis of, 243
Glass Manometer, Some Forms of, 437
Glazed Coffee, Resin in, Detection of, 275
Globulin, Serum, in Ammonium Sulphate Solutions, Coagulating-Points of Egg, Serum, and Milk Albumins, and of, 416
"Glow Reaction," Application of, to the Detection of the Platinum Metals, 434
Glucose Syrup, Sulphurous Acid in Estimation of, 543
Glucoside Reactions: Convallarin and Convallamarin, 289
(Glucosin) Gallisin, the So-Called, Occuring in Commercial Starch Syrup, Composition of, 543
Gluten, Wheat, Influence of Certain Vegetable Proteins on, 407
Glycerol, Crude, Analysis of, Report on, 314
————- Determination, Acetin Process for, Report on, 316
————————————-, Bichromate Process for, Report on, 319
—————, Estimation of, 289
————, Method of Saponification, Effect of, upon the Fatty Acids, 425
————- -Potassium Hydroxide, Estimation of the Reichert-Meissl Value of Fats by Means of, 542
Glycogen and Starch, the Separation of, Method for, 501

Glycuronic Acid, Small Quantities of, Detection of,.596
Glycyrrhizin and Sugars in Liquorice Powder and Extracts, Estimation of, 409
Goats' Milk; Composition of, and the Detection of Goats' Milk in Cows' Milk, 218
Gold and Silver, in Ores Containing Much Copper, Pot Assay of, 240
——, Estimation of, by Means of Hydrogen Peroxide, 34
——, Quantitative Analysis of, with the Use of Ether, 303
Goldenberg's Method, Estimation of Total Tartaric Acid by, 131
Gooch Crucible for Micro-Analysis, 180
Gorgonzola Cheese, Note on, 61
Government Laboratory, the Work of, for the Year Ending March 31, 1911, Report of the Principal Chemist upon, with Appendices, 571
———— Reports, 128, 479, 571
Grains, Brewers', Starch in, Estimation of, 500
Granular Form, Aluminium Hydroxide in, Precipitation of, 299
Graphite, Analysis of, 562
————, in Lubricants, Detection of, 356
Gravimetric Estimation of Tellurium, and Alkalimetric Estimation of Telluric Acid, 248
Grease, Heavy, Soap in, Estimation of' 559
———, Resistance to, Testing of Imitation Parchment Papers for, 597
Green Colour of Rapeseed Cakes, 411
Gum Arabic Pastilles, Analysis of, 344
—— from *Bombax Malabricum,* 356
—— Kino, Some Reactions of, 544
Gums from Northern Nigeria, 162
Gun-Cotton, Nitrates, etc., Nitrogen in, Estimation of, 243
————, Nitroglycerine, and the Like, Nitric Nitrogen in, Estimation of, 513

Hæmochromogen and the Spectroscopic Differentiation between Oxyhæmoglobin and Carbon Monoxide Hæmoglobin, 550
Hæmoglobin in Urine, the Failure of the Spectroscopic Test for, Hippuric Acid as a Cause of, 417
———— Preparations, 222
Halogens in Benzene Derivatives, the Estimation of, Use of Metallic Potassium in, 287
———— in Organic Compounds, Estimation of, 509
————, Other, Estimation of Iodine in Organic Compounds and its Separation from, 118
Halphen's Reagent for Colophony, Colour Reactions of Certain Resins with, 170
Hardness of Water, Estimation of: The Wartha-Pfeiffer Method and its Modification, 311
Heavy Grease, Soap in, Estimation of, 559
——— Liquids, Modified Separatory Funnel and Washer for, 89
Heller's Ring Test for the Detection of Albumin in Urine, etc., Modification of, 152
Hevea Latex, Some Analyses of, 6
Hexabromide Value of Oils, the Estimation of, Apparatus for, 607
Hexamethylenetetramine (Urotropine) in Urine, Estimation of, 285
Hippuric Acid as a Cause of the Failure of Spectroscopic Test for Hæmoglobin in Urine, 417
Histological Chemical Detection of Peroxydase, 75
Holder for Classen Dish, 180
Hollemann's Method, Improved, for the Estimation of Reverted Phosphoric Acid, 605
Honey, Analysis of, 276

Honey, Analysis of, with Special Reference to the Nitrogenous Constituents, 277
——, ——, Use of the Quantitative "Precipitin" Reaction in, 456
——, Bacteriological Estimation of, 505
——, —— Study of, 353
——, (Enzymes), Biological Examination of, 220
——, Examination of, by Fiehe's Reaction, 451
——, Formation, Chemistry of, 223
——, Invert Sugar in, Detection of, 586
——, Volatile Acids of, 277
——, Water in, Estimation of, 586
Hops, the Antiseptic Properties of, Valuation of, 65
——, Valuation of, by Analysis, 587
Hordenine, in Germinating Barley, Estimation of, 162
Hubener's Caoutchouc Tetrabromide, Notes on, 235
"Hughes' Reaction" of Potassium Iodide on Paper, and its Bearing on the Question of Acidity, 292
Human Body, Fluorine in, Distribution of, 75
Humus, in Soils, Estimating, 597
Husk and Germ in Cocoa Products, Quantity of, 214
——, in Cocoa Powder, Estimation of, 275
Hydrargyrometric Analyses, 357
Hypoiodites, the Formation of, and their Action on Sodium Thiosulphate, Notes on —A Source of Error in Certain Iodine Titrations, 132
Hypophosphite, Calcium, Analysis of, 605
Hypophosphorous Acid, Estimation of Copper by Means of, 302
Hydrated Silicic Acid in Clay, Determination of, 85
Hydrazine Hydrate, the Precipitation of Iron with, 521
—— Sulphate, Action of, on Nitrites, and a Method for the Estimation of Nitrite Nitrogen, 468
Hydride, Solid, of Antimony, Attempts to Prepare, 300
——, ——, — Arsenic, Detection of, 300
Hydrocarbon and Resin Oils in Other Oils, Detection of, 513
Hydrochloric Acid, Chlorine in, Electrolytic Determination of, with the Use of a Silver Anode and Mercury Cathode, 33
Hydrofluoric Acid, Preservation of Fruit Juices with, 218
Hydrogen, Active, in Organic Compounds, Quantitative Estimation of, by Means of Magnesium-Methyl Iodide, 117
——, ——, in Organic Molecules, Estimation of, 459
——, Carbon, Oxygen, and Nitrogen, Organic Substances Containing, New Method of Elementary Analysis of, 553
——, ——, ——, Nitrogen, and Sulphur, Analysis of a Substance Containing, 553
——, ——, ——, ——, Sulphur, Chlorine, Bromine, and Iodine, Analysis of a Substance Containing, 553
—— Cyanide, Traces of, the Detection of, 266
——, Delicate Test for, 34
——, in Gas Mixtures, Estimation of, 84
—— Peroxide, Estimation of Gold by Means of, 34
—— ——, Metals in Presence of, Influence of, on Blood Reagents, 352
—— —— Solutions, Acidity in, Estimation of, 82
—— ——, the Detection of Very Small Quantities of, Modified Cobalt Reaction for, 598
—— Sulphide, in Acid Solution, Rapid Qualitative Detection of Elements, the Sulphides of which are Precipitated by, 568

Hydrogen Sulphide, Qualitative Analysis without the Use of, 517
Hydrolyst, Determination of Sucrose in Beet Sugar Factory Products by Clerget's Process, Using Invertase as, 120
Hydroquinone, Estimation of, 163
Hydroxide, Aluminium, in Granular Form, Precipitation of, 299
———, Nickel, in Tannin Estimation, Use of, 516
Hydroxides, Alkali, or Bicarbonates, Alkali Carbonates in Presence of, Titration of, 362
Hydroxyl Groups, the Quantitative Estimation of, Use of Carbalkyloxy Derivatives for, 227
Hygienic Tests, Other, for Milk, Comparison of the Reductase Test with, 346
Hygroscopic, Poisonous, or Volatile Liquids, Apparatus for Storing and Measuring, 88
——— Substances or those Affected by Air, Apparatus for Filtering, 87

Ignition, Effect of, on Solubility of Soil Phosphates, 371
———, the Estimation of Carbon Dioxide in Carbonates and Nitrogen Pentoxide in Nitrates by loss on, Application of Sodium Paratungstate in, 526
Imitation Parchment Papers, Testing of, for Resistance of Grease, 597
Impact Machine (Drop Hammer), Sensitiveness Tests by the, 314
"Improvers," So-Called, the Addition of, to Flour, and the Bleaching of Flour, Report on, 254
Inactive Substances, Influence of, on the Rotation of Lævulose, 228
Incandescent Mantles, Testing of, 125
India, Travancore, Monazite Sand from, 523
———, Turpentine Oil from, 415
Indian Oils and Fats, Some, 108
Indicator, Methyl Red, as, Compared with Methyl Orange, 118
———, New, for the Estimation of "Titratable" Alkali in Blood, 415
———, Sensitive, 26
Indicators, Behaviour of Uranium Phosphates towards, 375
———, for Determining Affinity Values, Use of, 569
———, Phenolphthaleïn Derivatives and their Properties as, 169
Indigotin in the Presence of Starch, Estimation of, 290
Indophenol Oxydase of Animal Tissues, Estimation of, 550
Inert Gas, Precipitating, Filtering, and Drying in, Apparatus for, 313
Inflammable Gas and Vapour Detector, Automatic, New Type of, 528
INORGANIC ANALYSIS, ABSTRACTS, 30, 82, 123, 171, 236, 299, 362, 425, 466, 516, 559, 598
——— Phosphorus in Animal Tissue, Estimation of, 284
Insecticidal and Germicidal Values of Different Soaps, with or without Associated Active Agents, 505
Insecticides, Arsenic in, Estimation of, 237
INSTITUTE OF CHEMISTRY, 94, 193, 262, 444
Interpolation Method of Oil Analysis, 166
Intestines, Active Pepsin in, Detection of, by Means of Elastin, 551
Invert Sugar in Honey, Detection of, 586
Invertase as Hydrolyst, Determination of Sucrose in Beet Sugar Factory Products by Clerget's Process, Using, 120
———, in Wine, Detection of, 350
———, the Action of, Influence of the Viscosity on, According to the Concentration of Saccharose, 416
Iodic Acid, Detection and Estimation of Thorium by Means of. (Separation of Thorium from Scandium), 436
Iodide, Magnesium-Methyl, Quantitative Estimation of Active Hydrogen in Organic Compounds by Means of, 117

Iodides and Chlorides, Bromine in Presence of, Estimation of, 241
——————, Very Small Quantities of Bromine in Presence of, Estimation of, 365
———, Small Quantities of, in the Presence of Other Compounds, Estimation of, 241
Iodine, Action of, on Phenols, and their Volumetric Determination. Part I., 294
———, Alkali, and Silver, Volumetric Solutions of, Use of Sulphur Dioxide in Checking Strengths of, 248
———, Carbon, Hydrogen, Oxygen, Nitrogen, Sulphur, Chlorine, and Bromine, Analysis of a Substance Containing, 553
——— in Alcoholic Solutions, Estimation of, 235
——— in Organic Compounds, Estimation of, and its Separation from Other Halogens, 118
——— Solution, Titration of "Salvarsan" with, 350
——— Titrations, Certain, a Source of Error in—Notes on the Formation of Hypoiodites and their Action on Sodium Thiosulphate, 132
——— Values of Linoxyn, 24
——— ——— Oils, Measuring Flask for Use in Determining, 379
Iodoform, the Crystalline Form of, Reaction of Aromatic Inner Anhydrides Depending on their Influence on, 458
Iron Alloys, Estimation of Silicon in Vanadium and Molybdenum, and in their, 311
——, Aluminium, and Chromium, Precipitation of, by Ammonium Nitrite, 559
—— and Steel, Sulphur in, Volumetric Estimation of, 374
—————, Vanadium in, Colorimetric Method for, 473
—— and Vanadium, Separation of, by Means of Ether, 522
——————, Simultaneous Volumetric Estimation of, 475
——, Ferric, Volumetric Estimation of, by Permanganate after Reduction with Zinc, 521
——, Ferrous, in Silicate Rocks, Determination of, 602
——, Galvanised, Method for Testing, to Replace the Preece Test, 304
—— in Minerals, the Estimation of, the Influence of Pertitanic Acid on, 304
——, New Method of Separating, from Manganese, 604
—— Salts and Arsenic in Mineral Waters, Estimation of, 466
——, Separation of, from Aluminium, and New Test for Ferrous Oxide, 373
——, the Precipitation of, with Hydrazine Hydrate, 521
——, Tungsten, Molybdenum, and Vanadium, with High-Grade Alloys of, Estimation of Carbon and Sulphur in, 31
Ironstone, Chrome, Estimation of Chromium in, 428

Jackson's Candle Turbidimeter, Determination of Sulphur in Coal by Means of, 519
Jamaica Rum, Higher Alcohols Occurring in, 411
Jams, Analysis and Composition of, 144
——, etc., Starch Syrup in, Estimation of, 280
Jolles, the Method of, Polarimetric Estimation of Sugar by, 361
Juice, Liquorice, Analysis of, 410, 497
——, Prune, and Caramel in Vanilla Flavouring Extracts, Detection of, 281
——, Sugar-Cane, Acids in, Determination of, 594
——, Tobacco, and Extracts, Analysis of, and of Nicotine Salts, by the Method of Ulex, 545
—————, Concentrated, Estimation of Nicotine in, 106
Juices and Syrups, Sugar, Preparation and Constitution of Caramel and its Estimation in, 278
——, Fruit, Preservation of, with Hydrofluoric Acid, 218
——, ———, Salicylic Acid in, Estimation of, 411
——, Tobacco, Concentrated, Estimation of Nicotine in, 143

Kernels, Sweet Apricot, Composition of, 450
Ketones in Essential Oils, Estimation of, 514
Kino, Gum, Some Reactions of, 544
Knapp and Shrewsbury's Process for the Estimation of Cocoanut Oil, Examination of, 195
Kobert's Reagent as a Test for Salicylic Acid, 540

Laboratory Apparatus for the Production of a Constant Current of Ozonised Air, 479
———— Suction Pump, Simple, 532
Lac-Resin from Baroda, India, 171
————, Shellac, and Lac-Wax, Analytical Constants of, 119
Lactic Acid, Analysis of, 556
————, the Anhydride of, 235
Lactose in Urine and in Milk, Colorimetric Estimation of, 592
Lævulose, the Rotation of, Influence of Inactive Substances on, 228
Lamps, Safety, or Other Apparatus, for the Detection of Combustible Gases and Vapour, Methods of Testing, 528
Lard, Cocoanut Oil in, Estimation of, by Means of the "Ethyl Ester Value," 106
——, Small Quantities of Water in, Estimation of, 497
Latex, *Hevea*, Some Analyses of, 6
Lead Arsenate in the Products of Vines Treated with Arsenical Washes, Proportion of, 301
—— as Chromate, Volumetric Determination of, 602
——, Higher Oxides of, Valuation of, 366
—— in Alloys Containing Antimony and Tin, Estimation of, 367
—— Peroxide, Volumetric Estimation of, 467
Leaves, Coca, Analysis of, 274
Lecithin, 510
————, Estimation of, 510
————, in Oil, Estimation of, 166
Leguminous Plants Grown in Togo and German East Africa, Composition of the Seeds of, 345
Leuco-Malachite Green, Detection of Blood by Means of, 284
Lime, Separation of, from Large Quantities of Magnesia, 242
Linoxyn, Iodine Values of, 24
Linseed and Other Oils, Notes on, 278
———— Cake and Cocoanut Cake, the Effect of Feeding with, on the Composition of Butter-Fat, 445
———— Cakes and Linseed Meals, in Belgium, Adulteration of, 218
———— Oil, Analytical Examination of, 407
Liquid Fatty Acids, Separation of Solid from, 22, 114
————, Small Volumes of, Volumetric Analysis with, 185
Liquids and Solids, Vapour Pressures of, Static Method for Determining, 36
————, Aqueous, Automatic Extraction of, by Means of Specifically Lighter Solvents, 87
————, Heavy, Modified Separatory Funnel and Washer for, 89
————, Hygroscopic, Poisonous or Volatile, Apparatus for Storing and Measuring, 88
————, Mixed, Detection of Definite Compounds in, by its Means, and Direct Measurement of Changes of Vapour Pressure, 379
————, Organic, Purifying and Drying by Wiping, Method for, 26
————, Poisonous, Hygroscopic, or Volatile, Apparatus for Storing and Measuring, 88
————, the Density of, Determination of, 439
Liquorice Juice, Analysis of, 410, 497
———— Powder and Extracts, Glycyrrhizin and Sugars in, Estimation of, 409

Liquorice Powder, Compound, the Analytical and Microscopical Examination of, 198
Liquors, Tan, the Acidity of, Electrometric Method for the Estimation of, 121; Part II., 515
Lithium, Quantitative Estimation of, 175
Litmus, the Alcohol-Soluble Constituents of, 25
Logwood, Use of, in Qualitative Analysis, Estimation of Bismuth, 603
————, Valuation of, 26
Lophira alata (Banks), the Seeds of, Composition of the Fat from, 493
Lubricants, Graphite in, Detection of, 356
Lubricating Oils, Estimation of the Acidity of Fats and Oils, Particularly, 290
———— for Motor-Cars and Gas-Engines, 291
————, Mineral, Alcohol-Ether in, Proportion of Asphalt Insoluble in, 418
————, ————, of High Boiling-Point, Composition of, 461
Lubrication of Engines, Deposits Resulting from, 230

Machine, Impact (Drop Hammer), Sensitiveness Tests by, 314
Madeira, Preparation of, and its Composition, 410
Magnesia, Large Quantities of, Separation of Lime from, 242
———— Method for Estimation of Phosphoric Acid, Criticism of, 176
———— Mixture, Detection and Estimation of Arsenic Acid in Presence of Arsenious Acid by Means of, 30
Magnesium-Ammonium Phosphate, Estimation of Phosphoric Acid as, 434
———— and Calcium, Separation and Estimation of Barium in Presence of, by the Action of Acetyl Chloride and Acetone on the Mixed Chlorides, 426
———— as Oxide, Estimation of, 604
———— -Methyl Iodide, Quantitative Estimation of Active Hydrogen in Organic Compounds by Means of, 117
Malay States, Rare Earth-Containing Sands ("Amang"), from, 523
Malic Acid, the Determination of, Polarimetric Method for, and its Application in Cane and Maple Products, 498
Malt Analysis, the Method of, Improvement in, 146
Manganese, Electrolytic Estimation of, 177
————, Estimation of, as Oxide, 566
————, ————, by the Bismuth Method, 430
————, ————, by the Bismuthate Method, 305
————, ————, Rapid and Accurate Method for, 368
————, in Vanadium and Chrome-Vanadium Steels, Estimation of, 604
————, New Method of Separating Iron from, 604
————, Small Quantities of, Especially in the Ash of Plants or of Animal Organs, Estimation of, 369
————, the Volumetric Estimation of, Volhard's Method for, Waldemar Fischer's Modification of, and its Comparison with Other Well-known Methods, 52
————, Zinc, Cobalt, Nickel, Copper, and Cadmium, Precipitation of, from Ammoniacal Solutions by Means of Sodium Carbonate and Trimethylphenylammonium Carbonate, 599
Manometer, Glass, Some Forms of, 437
Mantles, Incandescent, Testing of, 125
Maple and Cane Products, Polarimetric Method for the Determination of Malic Acid, and its Application in, 498
Margarine, and Butter, Cocoanut Oil and Butter in, Some Methods of Estimating, Observations on, 333
————, German, the Poisonous Properties of Certain Brands of, Cause of, 216
————, the Analysis of, Notes on, 327
Marine Algæ, and Products Derived from Them, Relative Proportions of Arsenic in, 218

Marjoram, Spanish (*Thymus mastichina*, L.), Essential Oil of, 69
Mascarenhasia elastica, Rubber of, 159
Meals, Linseed, and Linseed Cakes, in Belgium, Adulteration of, 218
Measuring Flask for Use in Determining Iodine Values of Oils, 379
———— Radioactivity, Apparatus for, 380
———— the Degree of Vitiation of the Atmosphere, Method of, 363
Measurement, Direct, of Changes of Vapour Pressure and Detection of Definite Compounds in Mixed Liquids by its Means, 379
Meat and Yeast Extracts of Known Origin, Comparison of, 104
—— -Juices, Analysis of, 13
——, Potassium Nitrate in, Estimation of, 217
Melting-Point Determinations, 438
———— of Fats, Apparatus for the Estimation of, 569
Menthylglycuronic Acid, Preparation of, 505
Mercuric Chloride, Volumetric Estimation of, 34
Mercury Cathode and Silver Anode, Electrolytic Determination of Chlorine in Hydrochloric Acid with the Use of, 33
———— and Stationary Anode, Rapid Determinations and Separations by Means of, 83
————, Electrolytic Estimations with, Apparatus for, 529
—— in Urine, Detection of, 552
—— Pump for Rapidly Obtaining a High Vacuum, 89
—— Salts, Soluble, at Great Dilutions, Estimation of, 305
——, the Estimation of, Volumetric Method for, Depending on Reduction to Metal, 431
Metallic Acids, Action of Salicylic Action on, 567
Metals, Alkali, Estimation of, Elimination of Ammonium Salts Prior to, 30
——, in Presence of Hydrogen Peroxide, Influence of, on Blood Reagents, 352
——, Rapid Electro-Analytical Determination of, Apparatus for, 181
——, the Analysis of, Use of Ether in, 431
——, the Electrolytic Estimation of, Exact Method for, 370
——, the Platinum, Application of the "Glow Reaction" to the Detection of, 434
——, White, the Analysis of, Rapid and Accurate Method for, 606
Methyl Alcohol, Ethyl Alcohol in the Presence of, Detection of, 18
—— and Ethyl Alcohols and Formic Acid, Detection and Estimation of, 507
—— Orange, Methyl Red as an Indicator Compared with, 118
————, Use of, in the Colorimetric Estimation of Titanium, 310
—— Red as an Indicator Compared with Methyl Orange, 118
Methylpentosans and Pentosans in Cereals and Wood-Fungi, Estimation of, 460
Mexican Petroleum, Asphaltum from, 353
Micro-Analysis, Gooch Crucible for, 180
Microchemical Detection of Protein, 111
———— Test for Aluminium by Means of Caesium Chloride, 362
———————— Sodium, 372
Microchemistry, Use of Dimethylaminobenzene-azo-benzene Sulphonic Acid as a Reagent in, 232
Microdistillation Apparatus, 37
Microscopical and Analytical Examination of Compound Liquorice Powder, 198
———— Examination of Gambier, 344
———— ———— and Identification of Carbon, 428
———— Preparations, Combined Fixing and Staining Processes for, 569
Microtome Sections, Incineration of, 74
Milk, and Urine, Lactose in, Colorimetric Estimation of, 592
——, Catalase in, Estimation of, 284

Milk, Citric Acid in, Estimation of, 105
——, Condensed, Sweetened, Analysis of, 138
——, Cow's, on the Peroxydase of, and its Detection by Means of the *p*-Phenylenediamine Test, 222
——, ——, Goats' Milk in, the Detection of, and Composition of Goats' Milk, 218
——, ——, Oxydase and Reductase Occurring in, 16
——, Cryoscopic Examination of, 345
——, Egg, and Serum Albumins, Coagulating-Points of, and of Serum Globulin, in Ammonium Sulphate Solutions, 416
——, Evaporated, and Milk Products, Analyses of, 278
——, Fat in, the Estimation of, Wendler's Method for, 14
——, Goats', Composition of, and the Detection of Goats' Milk in Cows' Milk, 218
——, Nitrates in, Detection and Estimation of, by Means of the Diphenylamine-Sulphuric Acid Test, 67
——, ——— in, Estimation of, by Means of the Diphenylamine-Sulphuric Acid Test, 587
——, Other Hygienic Tests for, Comparison of the Reductase Test with, 346
——, Phosphorus in, the Gravimetric Estimation of, Note on, 579
—— Powders, Dried, Composition of, 279
—— Products, and Evaporated Milk, Analyses of, 278
—— Test, the "Neusal," 203
——, the Composition of, 390
——, the Degree of Accuracy with which Proteins can be Estimated in, by Aldehyde Titration, 9
——, the Examination of, Applicability of Recent Methods for, 347
——, the Phosphoric Acid in, Distribution of, 345
——, Very Small Quantities of, the Specific Gravity of, Determination of, 500
Milks, Condensed, an Inquiry as to, Report to the Local Government Board on; with Special Reference to their Use of Infants' Foods, 572
Mine, Coal in the, Sampling of, 527
Mineral Cylinder Oils, Dark, Tarry Substances in, Estimation of, 298
——— Grains, Errors in Determining Sizes of, and the Use of Surface Factors, 182
——— Lubricating Oils, Alcohol-Ether in, Proportion of Asphalt Insoluble in, 418
——— ————— —— of High Boiling-Point, Composition of, 461
(——— Matter) Fillers, in Rubber Mixtures, Estimation of, 558
——— Oil, in Wool-Grease Oleine, Detection of, 29
——— Oils and their Products, Origin of, Determination of, 167
——— Poisons, Method of Destroying Organic Matters in Testing for, 110
"——— Rubber" and Similar Products in Rubber Articles, Estimation of, 235
——— Waters, Arsenic and Iron Salts in, Estimation of, 466
Minerals, Differentiation of, by Means of Colour Reactions, 242
———, Iron in, the Estimation of, the Influence of Pertitanic Acid on, 304
———, Rare Earth, the Determination and Analysis of, Use of Sulphur Monochloride in, 567
Mines, the Gases Caused by Blasting in, Composition of, Report on, 321
Minute Quantities of Chromium, Colorimetric Estimation of, 238
——— ———— of Nitroglycerin, Estimation of, 556
Miscibility Curve, Analysis of Cod-Liver Oil by Means of its, 275
———— Curves, in the Examination of Essential Oils and Balsams, Use of, 556
Mixtures, Freezing, 127
———, Quantitative Analysis of, by Differences in the Specific Gravities of their Components, 306
Moisture, Use of Calcium Carbide for Determining, 112
Molecular Weight of Polysaccharides, the Estimation of, Colorimetric Method for, 360

Molecules, Organic, Active Hydrogen in, Estimation of, 459
Molybdenum and Vanadium, Estimation of Silicon in, and in their Iron Alloys, 311
——— Glance, Analysis of, 243
———, the Volumetric Estimation of, a New Method for, 98
———, Tungsten, and Vanadium, with Iron, High-Grade Alloys of, Carbon and Sulphur in, Estimation of, 31
Monazite Sand from Travancore, India, 523
——— Sands, Analysis of, 522
Monochloride, Sulphur, Use of, in the Determination and Analysis of Rare Earth Minerals, 567
Moroccan Olive Oil, 107
Morphine, Estimation of, by Extraction with Phenyl-ethyl Carbinol, 587
———, New Reaction of, 66
———, Quantitative Estimation of, 142
Mushroom, Soluble Basic Constituents of the, 347
Mutton-Bird Oil, Some Constants of, 290
Myrtenal and *d*-Perilla Aldehyde, the Occurrence of, in Nature: Essential Oil of "False Camphor-Wood," 289

Naphthalene in Crude Coal Gas, Estimation of, 510
Natural Gas and Fuel Oil, Specifications and Sampling of, 555
——— Vichy Salts, Characteristic Constituent of, 85
Neats-Foot Oil, 512
Neumann's Method, Estimation of Phosphoric Acid by, 525
"Neusal" Milk Test, 203
Neutralised Olive-Residuum Oil, Characteristics of, 116
Nickel and Cobalt, Gravimetric Estimation of, 598
——— in Admixture, Borax Bead Tests for, Efficiency of, 173
———, Rapid Determination of, by Means of the Gauze Cathode and Stationary Anode, 307
———, the Differentiation of, New Reaction for, 238
——— and Zinc in German Silver and Other Alloys, the Separation of, Method for, 524
——— Compounds, the Precipitation of, and Preparation of Spongy Nickel, 539
——— Hydroxide in Tannin Estimation, Use of, 516
——— in Steel, Determination of, Comparison of Methods for, 524
——— ——, Rapid Estimation of, 373
———, Spongy, Preparation of, and the Precipitation of Nickel Compounds, 539
———, Zinc, Manganese, Cobalt, Copper, and Cadmium, Precipitation of, from Ammoniacal Solutions by Means of Sodium Carbonate and Trimethylphenyl-ammonium Carbonate, 599
Nicotiana tabacum, Green Plants of, and Tobacco, Nicotine in, Accurate Estimation of, 348
Nicotine, Estimation of, in the Presence of Pyridine Bases, 452
——— in Concentrated Tobacco Juice, Estimation of, 106, 143
——— in Nicotine Solutions and Tobacco Extracts, Estimation of, 544
———, in Tobacco and in Green Plants of *Nicotiana tabacum*, Accurate Estimation of, 348
———, ——— Extracts, Estimation of, 348
——— ——— Polarimetric Estimation of, 279
——— ——, the Estimation of, New Method for, 280
——— Salts, Analysis of, and of Tobacco Juice and Extracts, by the Method of Ulex, 545
Niger-Seed Oil, 358
Nigeria, Northern, Gums from, 162

Nitrate and Nitrite Nitrogen in Commercial Calcium Nitrate and Nitrite, 524
———, Estimation of, 467
———, Gasometric Estimation of, by Schloesing's Method, and of Nitrite by Piccini's Method, Some Sources of Error in, 525
Nitrates and Nitrites, in Water, Detection of, 432
———, Estimation of, by the Phenol Sulphonic Acid Method, 84
———, Gun-Cotton, etc., Nitrogen in, Estimation of, 243
——— in Milk, Detection and Estimation of, by Means of the Diphenylamine-Sulphuric Acid Test, 67
——————, Estimation of, by Means of the Diphenylamine-Sulphuric Acid Test, 587
——— in Water, Estimation of, by a Sulpho-Salicylic Reagent, 85
———, ———, the Estimation of, Study of the Phenolsulphonic Acid Method for. III. Chief Sources of Error, 244
———, Nitrogen Pentoxide in, and Carbon Dioxide in Carbonates, the Estimation of, by Loss on Ignition, Application of Sodium Paratungstate in, 526
———, the Estimation of, Rapid Method for, 468
Nitre, Norwegian, Analysis of, and of Calcium Cyanamide, 561
Nitric Acid, and Nitrous Acid in Water, Detection and Estimation of, 433
—————, Decomposition of the Bromide by, Estimation of Rubber as Tetrabromide, 361
—————, in Nitrocellulose, Nitroglycerol, and Other Compounds, Volumetric Determination of, 34
————— in Vegetable Products, Quantatitive Estimation of, 163
——— and Nitrous Acids, the Diphenylamine Test for, Modification of, 370
——— Nitrogen in Gun-Cotton, Nitroglycerine, and the Like, Estimation of, 513
——— Oxide, Estimation of, and its Behaviour towards Water, 433
Nitrite, Ammonium, Precipitation of Aluminium, Chromium, and Iron by, 559
——— and Nitrate, Commercial Calcium, Estimation of Nitrate and Nitrite Nitrogen in, 524
———, Gasometric Estimation of, by Piccini's Method, and of Nitrate by Schloesing's Method, Some Sources of Error in, 525
——— in Sewage Effluents and Water, the Amount of, the Determination of, and of the Amount of Dissolved Oxygen Absorbed by Sewage Effluents Containing Nitrites, 393
Nitrites, Action of Hydrazine Sulphate on, and a Method for the Estimation of Nitrite Nitrogen, 468
——— and Nitrates in Water, Detection of, 432
———, in Water, Detection of, 370
———, New Method of Estimating, 307
Nitrocellulose, Nitroglycerol, and Other Compounds, Nitric Acid in, Volumetric Determination of, 34
Nitrogen and Carbon in Organic Compounds, Estimation of, 593
———, Carbon, Hydrogen, and Oxygen, Organic Substances Containing, New Method of Elementary Analysis of, 553
——————, Oxygen, and Sulphur, a Substance Containing, Analysis of, 553
—————————, Sulphur, Chlorine, Bromine, and Iodine, a Substance Containing, Analysis of, 553
——— in Liquids, Estimation of, 421
———, in Nitrates, Gun-Cotton, etc., Estimation of, 243
———, Nitric, Gun-Cotton, Nitroglycerine, and the Like, Estimation of, 513
Nitrate and Nitrite in Commercial Calcium Nitrate and Nitrite, Estimation of, 524

Nitrogen, Nitrite, a Method for the Estimation of, and Action of Hydrazine Sulphate on Nitrites, 468
———, Organic, Available, Bacteriological Method of Estimating, 219
Nitrogenous Constituents, Analysis of Honey, with Special Reference to the, 276
Nitroglycerine, Gun-Cotton, and the Like, Nitric Nitrogen in, Estimation of, 513
Nitroglycerin, Minute Quantities of, Estimation of, 556
Nitroglycerol, Nitrocellulose, and Other Compounds, Nitric Acid in, Volumetric Determination of, 34
Nitrosite, Caoutchouc, and its Analytical Application, 287
Nitrous Acid, and Nitric Acid, in Water, Detection and Estimation of, 433
——— and Nitric Acids, the Diphenylamine Test for, Modification of, 370
——— Oxide, in the Gases Produced by Denitrifying Organisms, Occurrence, Detection, and Estimation of, 591
—————. Chemistry of Anæsthetics, III., 503
Normal Weight of Dextrose, 78
——— Yellow Pigmentary Substances of Urine, Relation of, to the Diazo-Reaction, and a Colorimetric Method for Estimating Urochrome and Urochromogen, 223
Norwegian Nitre, Analysis of, and of Calcium Cyanamide, 561
Nuclease, the Enzymatic Activity of, Estimation of, 109
Nutritive Value of Bread made from Different Varieties of Flour, Report to the Local Government Board on, 479

Occlusion, Phenomenon of, in Precipitates of Barium Sulphate, and its Relation to the Exact Determination of Sulphates, 426
Ochres, Bismuth, from California, 174
Oil, African Sandal-Wood, Adulterant of, 146
—— Analysis, Interpolation Method of, 166
—— and Tincture of Cantharides, Cantharidine in, Estimation of, 404
——, Cocoanut, and Butter, in Butter and Margarine, Some Methods of Estimating, Observations on, 333
——, ————, in Butter, Detection of, by Means of Ewer's Method, 451
——, ————, ————, ————, ———— Fendler's Method, 451
——, ————, in Chocolate, the Detection of, Note on, 275
——, ————, in Lard, Estimation of, by Means of the "Ethyl Ester Value," 106
——, ————, Shrewsbury and Knapp's Process for the Estimation of, Examination of, 195
——, Cod-Liver, Analysis of, by Means of its Misability Curve, 275
——, Creosote, and Creosote Baths (Dips), the Dimethyl Sulphate Test for, 585
——, Essential, of Orange Flowers, 67
——, ————, of *Santolina Chamæcyparissus*, 589
——, ————, of *Schinus molle* (L.), 68
——, ————, of Spanish Marjoram (*Thymus mastichina*, L.), 69
——, Fatty, and Wax, Obtained from Coffee Berries, 106
—— from Seeds of the Ash-Tree, 357
—— from the Seeds of *Xanthophyllum lanceolatum*, 406
——, Fuel, and Natural Gas, Specifications and Sampling of, 555
——, Fusel, in Brandy, Detection of, 216
——, Lecithin in, Estimation of, 166
——, Linseed, Analytical Examination of, 407
——, Mineral, in Wool-Grease Oleine, Detection of, 29
——, Mutton-Bird, Some Constants of, 290
——, Neats-Foot, 512
——, Niger-Seed, 358
——, Olive, Moroccan, 107

Oil, Olive-Residuum, Neutralised, Characteristics of, 116
—, ——, Wines, and Vinegar, Portuguese Standards for. (Extract from the Official Methods of Analysis), 502
—. Para Rubber-Seed, Composition of, 491
—, Rice, 357
—, Soya Bean, 358
—, ————, Composition of, 513
—, Spanish, of Turpentine, 589
—, Train, in Oils, Fats, and Soaps, Detection of, 359
—, Turpentine, Analysis of, 546
—, ————, from India, 415
—, Wood-Turpentine, Turpentine Oil, and Rosin Essence, Reaction of, 107
Oils, Almond and Apricot Kernel, Note on, 263
— and Fats, Analysis of, 164
————, Edible, Colour Reactions of, 406
————, Particularly Lubricating Oils, the Acidity of, Estimation of, 290
————, Some Indian, 108
————, the Bromine Value of, Estimation of, Primary and Secondary Bromine Values, 19
————, Vegetable, Some Little-known, Characteristics of, 21
—, Apricot Kernel and Almond, Note on, 263
—, Certain, Characteristics of, 21
—, Conifer Fatty, Characteristics of, 511
—, Dark Mineral Cylinder, Tarry Substances in, Estimation of, 298
—, Essential, and Balsams, the Examination of, Use of Miscibility Curves in, 556
—, ———, and Philippine Terpenes, 15
—, ———, Estimation of, 288
—, ———, Ketones in, Estimation of, 514
—, Fat, and Soaps, the Total Fatty Acids in, Estimation of, 115
—, Fats, and Soaps, Train Oil in, Detection of, 359
—, Fats, and Waxes, the Saponification Value of, Estimation of, Use of Propyl Alcohol, 290
— from Papilionaceous Plants, Characteristics of, 231
—, Fatty, Rosin in, Examination of, 557
—, Iodine, Values of, Measuring Flask for Use in Determining, 379
—, Linseed, and Other, Notes on, 278
—, Lubricating, Estimation of the Acidity of Fats and Oils, Particularly, 290
—, ————, for Motor-Cars and Gas-Engines, 291
—, ————, Mineral, of High Boiling-Point, Composition of, 461
—, Mineral, and their Products, Origin of, Determination of, 167
—, ———, Lubricating, Alcohol-Ether in, Proportion of Asphalt Insoluble in, 418
— of Turpentine, Analysis of, II., 150
—, Resin, and Hydrocarbon in Other Oils, Detection of, 513
—, Rosin, in French Wool-Grease Oleines, Detection of, 30
—, Tar, and Creosote, Available for Wood Preservation, Characters of, 228
—, the Hexabromide Value of, the Estimation of, Apparatus for, 607
—, the Specific Gravity of, Estimation of, 557
—, Wool, Unsaponifiable Matter in, Estimation of, 168
Oleic Acid, Separation of, from Stearic and Palmitic Acids, 23
Oleine, Wool-Grease, Mineral Oil in, Detection of, 29
Oleines, Wool-Grease, Examination of, 29
———, ————, French, Rosin Oils in, Detection of, 30
Olive Oil, Moroccan, 107

Olive Oil, Wines, and Vinegar, Portuguese Standards for. (Extract from the Official Methods of Analysis), 502
—— -Residuum Oil, Neutralised, Characteristics of, 116
(Onchocerciasis) a Parasitic Condition Met with in Australian Beef, 129
Opium, Codeine in, the Determination of, 489
Opsonin Reaction, 455
Orange Flowers, Essential Oil of, 67
Ores, Bog-Iron, Analysis of, 123
—— Containing Much Copper, Gold, and Silver in, Pot Assay of, 240
——, Refractory Tin, Simple Method of Decomposing, 309
——, Tin in, Estimation of, 527
ORGANIC ANALYSIS, ABSTRACTS, 17, 75, 112, 157, 224, 287, 353, 417, 457, 507, 553, 592
Organic Bodies, Influence of Potassium Permanganate upon, 232
—— Liquids, Purifying and Drying by Wiping, Method for, 26
—— Matter, Incineration in Presence of, Causes of Loss of Phosphoric Acid on, 360
————, Method of Destroying, by Means of Bromine, Particularly Applicable to Toxicological Analysis, 592
—— Matters, Changes which Mercury Compounds Undergo when in Contact with, 154
————, Method of Destroying, in Testing for Mineral Poisons, 110
—— Molecules, Active Hydrogen in, Estimation of, 459
—— Nitrogen, Available, Bacteriological Method of Estimating, 219
—— Substances, Action of Permanganate on, 422
———— in Water, Estimation of, by Means of Permanganate, 502
———— the Estimation of Carbon in, Wet Method for, 354
Organisms, Denitrifying, the Gases Produced by Occurrence, Detection, and Estimation of Nitrous Oxide in, 591
Osmotic Pressure and Electrical Conductivity, Application of, in the Examination of Soils, 184
Oven, Drying, Constant Temperature, New Form of, 126
Ovolecithin, Characteristic Colour Reaction of, 460
Oxidation of Ferrous Salts, 467
Oxide, Estimation of Manganese as, 566
——, Ferric, in Cement, Rapid Estimation of, 563
————, Separation of Alumina from, 82
—— Ferrous, in Silicates, the Titration of, by the Pebal-Dölter Method, Note on, 372
————, New Test for, and the Separation of Iron from Aluminium, 373
—— Magnesium as, Estimation of, 604
—— Nitric, Estimation of, and its Behaviour towards Water, 433
—— Nitrous, Chemistry of Anæsthetics, III., 503
————, in the Gases Produced by Denitrifying Organisms, Occurrence, Detection, and Estimation of, 591
——, Phosphoric, in Basic Slag, Citrate-Solubility of, 32
Oxides, Higher, of Lead, Valuation of, 366
Oxidising Agents, the Activity of, Estimation of, by Means of the Quinonoid Imonium Salts of Benzidine, 233
—— Substances in Animal Organs, Colorimetric Detection of, 152
Oxydase and Reductase Occurring in Cow's Milk, 16
——, Indophenol, of Animal Tissues, Estimation of, 550
Oxygen, Carbon, Hydrogen, and Nitrogen, Organic Substances Containing, New Method of Elementary Analysis of, 553
——, Carbon, Hydrogen, Nitrogen, and Sulphur, Analysis of a Substance Containing, 553

Oxygen, Carbon, Hydrogen, Nitrogen, Sulphur, Chlorine, Bromine, and Iodine, Analysis of a Substance Containing, 553
———, Commercial, Analytical Examination of, 469
———, Dissolved, Absorbed by Sewage Effluents Containing Nitrites, the Determination of the Amount of, and of the Amount of Nitrite in Sewage Effluents and Water, 393
———, ———, Estimation of, 245
——— in Air, Volumetric Estimation of, 569
Oxyhæmoglobin, and Carbon Monoxide Hæmoglobin, the Spectroscopic Differentiation between, and Hæmochromogen, 550
Ozone, Estimation of, by Means of Alkaline Potassium Iodide Solution, 525
Ozonised Air, a Constant Current of, Laboratory Apparatus for the Production of, 479

Paints, Consistency of, by the Stormer Viscosimeter, 609
———, New Accelerated Test for, 608
Palmitic and Stearic Acids, Separation of Oleic Acid from, 23
Paper, Animal Sizing in, Detection of. Differentiation of Gelatin and Casein, 290
———, "Hughes' Reaction" of Potassium Iodide on, and its Bearing on the Question of Acidity, 292
———, Reagent, for the Volumetric Estimation of Zinc, 126
Papers, Imitation Parchment, Testing of, for Resistance to Grease, 597
Papilionaceous Plants, Oils from, Characteristics of, 231
Para Rubber-Seed Oil, Composition of, 491
Paraffin, Beeswax, Spermaceti, etc., Fat in, Detection of, 496
Pastilles, Gum Arabic, Analysis of, 344
Pathological Urine, Creatine in, Direct Estimation of, 504
———————, Fat in, Estimation of, 417
"Pearl Coating" of Pills, Note on, 389
Pears, Apples, Cider, and Perry, Tartaric Acid, Estimation of, 545
Pebal-Dölter Method, the Titration of Ferrous Oxide in Silicates by, Note on, 372
Pentosans and Methylpentosans in Cereals and Wood-Fungi, Estimation of, 460
———— and Pentoses, the Quantitative Analysis of, and its Practical Applications, Researches on, 293
————, Estimation of, with the Aid of Fehling's Solution, 161
———— of Certain Wood-Fungi, 162
————, the Estimation of, the Furfural Method for, Modification of, 161
Peppercorns, Adulteration of, 349
Pepper, the Analysis of, New Constant in, 147
Pepsin, Active, in the Intestines, Detection of, by Means of Elastin, 551
———, the Valuation of, Comparative Studies on, 155
Peptones, Commercial, Certain, Physical Constants and Analyses of, 111
———— or Peptides, and Amino-Acids, Differentiation between, 593
Perborates, and Similar Compounds, Detection of, 470
Perchlorate, Potassium, in Potassium Chlorate, Estimation of, 248
Perchlorates, Chlorides, and Chlorates, in Presence of Each Other, Estimation of, 365
"Perhydrol" for the Estimation of Total Sulphur in Coal Gas, 309
d-Perilla Aldehyde, and Myrtenal, Occurrence of, in Nature: Essential Oil of "False Camphor-Wood," 289
Permanganate, Action of, on Organic Substances, 422
——————, Estimation of Organic Substances in Water by Means of, 502
——————, —————— Very Small Amounts of Calcium by Means of, 174
——————, Potassium, Influence of, upon Organic Bodies, 232
——————, —————, Separation of Cerium by, 429

Permanganate Solutions, Ferric Sulphate as Standard for, 520
—————, Volumetric Estimation of Ferric Iron by, after Reduction with Zinc, 521
Peroxide, Hydrogen, Estimation of Gold by Means of, 34
————, ————, Metals in Presence of, Influence of, on Blood Reagents, 352
————, ————, Solutions, Estimation of Acidity in, 82
————, ————, the Detection of Very Small Quantities of, Modified Cobalt Reaction for, 598
————, Lead, Volumetric Estimation of, 467
Peroxydase, Histological-Chemical Detection of, 75
———— of Cow's Milk, and its Detection by Means of the *p*-Phenylenediamine Test, 222
Perry, Apples, Pears, and Cider, Tartaric Acid in, Estimation of, 545
Pertitanic Acid, the Influence of, on the Estimation of Iron in Minerals, 304
Petroleum Asphalt, Technical Analysis of, 294
———— in Turpentine, Henry C. Frey's Method of Estimating, Note on, 137
————, Mexican, Asphaltum from, 353
———— Pitch, in Fat Pitch, Detection of, 462
———— Spirit, Extinguishing the Flame of, 252
Phenol and *p*-Cresol in the Presence of Each Other, Estimation of, 79
——— and Salicylic Acid, Sensitive Test for, 295
——— Sulphonic Acid Method, Estimation of Nitrates by, 84
Phenolphthaleïn Derivatives and their Properties as Indicators, 169
——————, Volumetric Estimation of, 14
Phenols, Action of Iodine on, and their Volumetric Determination. (Part I.), 294
Phenolsulphonic Acid Method for the Estimation of Nitrates in Water, Study of. III. Chief Sources of Error, 244
Phenolsulphonic Acid Method for the Estimation of Nitrates in Water, Study of. IV. A Modified Method, 244
Phenyl-ethyl Carbinol, Estimation of Morphine by Extraction with, 587
p-Phenylenediamine Test, on the Peroxydase of Cow's Milk, and the Detection by Means of the, 222
Philippine Terpenes and Essential Oils, 15
Phosphate, Magnesium-Ammonium, Estimation of Phosphoric Acid as, 434
Phosphates, Soil, Solubility of, Effect of Ignition on, 371
—————, Titration of, 473
—————, Uranium, Behaviour of, towards Indicators, 375
Phosphoric Acid, as Magnesium-Ammonium Phosphate, Estimation of, 434
——————, Colorimetric Estimation of, 525
——————, ————— Method of Estimating, 307
——————, Estimation of, by Neumann's Method, 525
——————, in Milk, Distribution of, 345
——————, Loss of, on Incineration in Presence of Organic Matter, Causes of, 360
——————, Magnesia Method for Estimation of, Criticism of, 176
——————, Reverted, the Estimation of, Improved Hollemann's Method for, 605
—————— Soluble in 2 per cent. Citric Acid Solution, Volumetric Estimation of, 308
——————, Volumetric Estimation of, 472
—————— and Vanadic Acids, in Presence of Each Other, Estimation of, and Precipitation of Vanadic Acid as Silver Vanadate, 35
—— Oxide, in Basic Slag, Citrate Solubility of, 32
Phosphorus and Sulphur in Organic Substances, Estimation of, 81
———— in Milk, the Gravimetric Estimation of, Note on, 579

Phosphorus, Inorganic, in Animal Tissues, Estimation of, 284
———— in Small Amounts, Titrimetric Estimation of, 246
Phosphotungstic Acid Precipitation of Proteins, 461
Physical Constants and Analyses of Certain Commercial Peptones, 111
Piccini's Method, Gasometric Estimation of Nitrite by, and of Nitrate by Schloesing's Method, Some Sources of Error in, 525
Pigmentary Substances, Normal Yellow, of Urine, Relation of, to the Diazo-Reaction, and a Colorimetric Method for Estimating Urochrome and Urochromogen, 223
Pill, Blaud's, the Composition of, Note on, 387
Pills, the "Pearl Coating" of, Note on, 389
Pitch, Fat, Petroleum Pitch in, Detection of, 462
Plants, Leguminous, Grown in Togo and German East Africa, the Seeds of, Composition of, 345
——— or Animal Organs, the Ash of, Estimation of Small Quantities of Manganese, Especially in, 369
Plaster of Paris, Testing, 246
Platinum and Tin, Separation of, 177
————, Assay of, 605
———— Chloride, Precipitation of Cocaine Solutions with, 354
———— Metals, the Detection of, Application of the "Glow Reaction" to, 434
Poisonous "Cardamon" (Maratti) Fat Employed in the Manufacture of "Backa-Margarine," Nature of, 542
————, Hygroscopic, or Volatile Liquids, Apparatus for Storing and Measuring, 88
———— Properties of Certain Brands of German Margarine, Cause of, 216
———— Ricinus Constituents in Fodder-Stuffs, Detection of, 506
Poisons, Mineral, Method of Destroying Organic Matters in Testing for, 110
Polarimetric Estimation of Sugar by the Method of Jolles, 361
Polysaccharides, the Molecular Weight of, Colorimetric Method for the Estimation of, 360
Porosity of Earthenware Filtering Bougies, Testing the, 16
Portland Cement, Uniform Methods of Analysis for, 247
Portuguese Standards for Wines, Vinegar, and Olive Oil. (Extract from the Official Methods of Analysis), 502
Pot Assay of Gold and Silver in Ores Containing Much Copper, 240
Potable Spirits, Analysis of, and its Bearing on the Definition of Whisky (Report), 441
Potash Bulb, 86
Potassium as Potassium Platini-Chloride, Estimation of, 178
———— Bitartrate, and Calcium Tartrate, Separate Estimation of, 234
———— Chlorate, Potassium Perchlorate in, Estimation of, 248
———— Chloriplatinate, Reduction of, 526
———— Cyanide, in Presence of Potassium Ferrocyanide, Titration of, 460
———— Ferrocyanide, Pure, Preparation of, and its Analytical Control, 564
————————, Titration of Potassium Cyanide in Presence of, 460
———— Iodide, "Hughes' Reaction" of, on Paper, and its Bearing on the Question of Acidity, 292
———————— Solution, Alkaline, Estimation of Ozone by Means of, 525
———— Metallic, in the Estimation of Halogens in Benzene Derivatives, Use of, 287
———— Nitrate in Meat, Estimation of, 217
———— Perchlorate, in Potassium Chlorate, Estimation of, 248
———— Permanganate, Influence of, upon Organic Bodies, 232
————————, Separation of Cerium by, 429
————, Small Quantities of, Detection of, as the Cobalti-Nitrite, 606

Powder and Extracts, Liquorice, Glycyrrhizin and Sugars in, Estimation of, 409
———, Cocoa, Husk in, Estimation of, 275
———, Compound Liquorice, the Analytical and Microscopical Examination of, 198
———, Smokeless, Camphor in, Estimation of, 420
———————, Stability Tests of, 295
Powders, Dried Milk, Composition of, 279
Precipitates of Barium Sulphate, Phenomenon of Occlusion in, and its Relation to the Exact Determination of Sulphate, 426
Precipitating, Filtering, and Drying, in an Inert Gas, Apparatus for, 313
Precipitin Reaction, 456
———————, the Quantitative, Use of, in Honey Analysis, 456
Preece Test, Method for Testing Galvanised Iron to Replace the, 304
Preparations, Hæmoglobin, 222
————, Microscopical, Combined Fixing and Staining Processes for, 569
Pressure, Osmotic, and Electrical Conductivity, Application of, in the Examination of Soils, 184
———, Reduced, Rapid Electro-Analysis under, 601
———, Vapour, Direct Measurement of Changes of, and Detection of Definite Compounds in Mixed Liquids by its Means, 379
Pressures, Vapour, of Solids and Liquids, Static Method for Determining, 36
Propyl Alcohol, Use of. Estimation of the Saponification Value of Oils, Fats, and Waxes, 290
Protein, Microchemical Detection of, 111
Proteins, an Inner Anhydride Reaction of, 514
———, and their Derivatives, the Detection of, Use of Triketohydrindene Hydrate for, 514
———, Certain Vegetable, Influence of, on Wheat Gluten, 447
———, Colour Reaction of, with Sodium Nitroprusside, 235
———, Phosphotungstic Acid Precipitation of, 461
———, the Analysis of, a Method for, and Estimation of Amino Groups in Amino Compounds and in Urine, 457
———, the Precipitation of, Experiments on, 295
———, Urine, and Enzymes, the Chemistry of, Quantitative Estimation of Aliphatic Amino Groups, and its Application in, 17
Proteolytic Enzymes, the Detection of, Use of Elastin in, 590
Prune Juice and Caramel in Vanilla Flavouring Extracts, Detection of, 281
Pulp of *Cassia fistula*, Composition of, 214
Pump, Mercury, for Rapidly Obtaining a High Vacuum, 89
——, Modified Boltwood Vacuum, 128
——, Suction, Simple Laboratory, 532
Pyridine and Ammonia, Separation and Estimation of, 508
——— Bases, Nicotine in the Presence of, Estimation of, 452
——— in Vinegar, Estimation of, 414
Pyrites and Burned Pyrites, Copper in, Estimation of, 366
———, Arsenic in, Estimation of, 362
Pyrrol, Quantitative Estimation of Acetaldehyde by Means of, 157

Qualitative Analysis, Decomposition of Silicates by Fusion with Boric Acid as Applied to, 371
———————, Use of Logwood in. Estimation of Bismuth, 603
——————— without the Use of Hydrogen Sulphide, 517
Quantitative Fermentation Experiments, Flask for, 568
———— Separation by Means of "Cupferron," 520
Quinine, in Durgs, etc., Volumetric Estimation of, 143

Quinine, the Estimation of, as the Acid Citrate in Certain Organic Liquids, 396
Quinones, Volumetric Estimation of, by Means of Titanium Trichloride, 80
Quinonoid Imonium Salts of Benzidine, Estimation of the Activity of Oxidising Agents by Means of, 233

Racemic Acid as a Reagent (Precipitant of Calcium and Strontium), 365
Radioactivity, Apparatus for Measuring, 380
——— of Radium, the Measurement of, International Standard Units for, 35
Rapeseed Cakes, Green Colour of, 411
Raw Rubber, the Analytical Methods for, Critical Investigations on, 27
———, the Valuation of, Comparative Tests for, Proposed Uniform Method of Carrying out, 423
Raw Sugars and Other Products, the Polarisation of, upon Quartz Wedge Saccharimeters, Use of Temperature Corrections in, 297
Rays, Ultra-Violet, Sterilisation of Drinking Water by Means of, 457
Reagent Paper for the Volumetric Estimation of Zinc, 126
———, Schardinger's, Behaviour of, towards Colostrum, 221
———, Sulpho-Salicylic, Estimation of Nitrates in Water by, 85
Reagents, Blood, Influence of Metals in Presence of Hydrogen Peroxide on, 352
Recorder, Multiple Carbon Dioxide, 252
Recording Thermo-Electric Calorimeter, 312
Reducing Sugars, Estimation of, 298
Reductase and Oxydase Occurring in Cow's Milk, 16
——— Test, Comparison of, with Other Hygienic Tests for Milk, 346
Reduced Pressure, Rapid Electro-Analysis under, 601
Refined Copper, Exact Electrolytic Assay of. I. Standard Method, 172
Refractometer, Estimation of the Salinity of Sea Water by Means of the, 311
Refractometric Examinations, Thermostat for, 89
Refractory Tin Ores, Simple Method of Decomposing, 309
Regulator, Gas, for Thermostats, 127
Regulators, Gas-Pressure, Simple Forms of, 477
Reichert-Meissl Value of Fats, Estimation of, by Means of Glycerol-Potassium Hydroxide, 542
Report, Annual, of the Dominion Laboratory, Wellington, New Zealand (1909), 91
———, Composition of the Gases Caused by Blasting in Mines, 321
——— (Government Paper, Western Australia, 1911). Analysis of Potable Spirits, and its Bearing on the Definition of Whisky, 441
———, Metropolitan Water Board, 185
——— of the Malt Analysis Committee (Coloured Malts and Caramel), 1910, to the Council of the Institute of Brewing, 90
——— of the Principal Chemist upon the Work of the Government Laboratory for the Year ending March 31, 1911, with Appendices, 571
——— on a Parasite Condition (Onchocerciasis) Met with in Australian Beef, 129
——— on the Bleaching of Flour and the Addition of So-called "Improvers" to Flour, 254
——— on the Chemical Changes Produced in Flour by Bleaching, 254
——— on the Presence of Calcium Sulphate in Baking-Powder and Self-Raising Flour, 255
——— on the Solubility, Extraction, and Reactions of Certain Colouring Matters Used in Foods, 186
——— on the Work of Inspectors of Foods, 128
——— on the Work of the Laboratories, Survey Department, Egypt (1910), 532
——— to the Government of Western Australia, 1911, 321

Report to the Local Government Board on the Nutritive Value of Bread Made from Different Varieties of Flour, 479
———, United States Department of Agriculture, Bureau of Chemistry, Circular, 186
Residues from the Distillation of Coal Tar, Rosin in, Estimation of, 463
Resin, Dammar, Different Varieties of, Characteristics of, 422
—— in Glazed Coffee, Detection of, 275
——, Lac, from Baroda, India, 171
—— Oils and Hydrocarbon in Other Oils, Detection of, 513
Resins, Certain, Colour Reactions of, with Halphen's Reagent for Colophony, 170
Rice Oil, 357
Ricinus Constituents, Poisonous, in Fodder-Stuffs, Detection of, 506
Ripe and Unripe Bananas, Composition of, 274, 541
Rocks, Silicate, Ferrous Iron in, Determination of, 602
Roquefort Cheese, Composition of, 542
Rosin Essence, Turpentine Oil and Wood Turpentine Oil, Reaction of, 107
—— in Fatty Oils, Estimation of, 557
—— in the Residues from the Distillation of Coal Tar, Estimation of, 463
—— Oils in French Wool-Grease Oleines, Detection of, 30
Rubber Articles, "Mineral Rubber," and Similar Products in, Estimation of, 235
——————, Vulcanised, Caoutchouc in, Direct Estimation of, 113
———, Cearà, 424
———, Estimation of, as Tetrabromide. Decomposition of the Bromide by Nitric Acid, 361
——— Goods, Vulcanised, Caoutchouc in, Direct Estimation of, 75
——— Mixtures, Fillers (Mineral Matter) in, Estimation of, 558
——— of *Mascarenhasia elastica*, 160
———, Raw, the Analytical Methods for, Critical Investigations on, 27
the Valuation of, Proposed Uniform Method of Carrying Out Comparative Tests for, 423
--Seed Oil, Para, Composition of, 491
Stoppers, Substitute for, 610
Rum, Jamaica, Higher Alcohols Occurring in, 411
Rye and Wheat Bran, Customs Examination of, 350

Saccharimeters, Quartz Wedge, the Polarisation of Raw Sugars and Other Products upon, Use of Temperature Corrections in, 297
Saccharose, the Concentration of, Influence of the Viscosity on the Action of Invertase According to, 416
Safety Lamps, or Other Apparatus, for the Detection of Combustible Gases and Vapour, Methods of Testing, 528
Salicylate, Bismuth, Examination of, 342
Salicylic Acid, a Test for, Kobert's Reagent as, 540
—————, Action of, on Metallic Acids, 567
————— and Phenol, Sensitive Test for, 295
Detection of, 463
– in Fruit Juices, Estimation of, 411
Salinity of Sea Water, Estimation of, by Means of the Refractometer, 311
Salts, Aluminium, Free Acid and Basic Alumina in, Estimation of, 236
——, Ammonium, Elimination of, Prior to Estimation of Alkali Metals, 30
——, Iron, and Arsenic, in Mineral Waters, Estimation of, 466
——, Mercuric, Soluble, at Great Dilutions, Estimation of, 305
——, Natural Vichy, Characteristic Constituent of, 85
——, Nicotine, Analysis of, and of Tobacco Juice and Extracts, by the Method of Ulex, 545

Salts, Quinonoid Imonium, of Benzidine, Estimation of the Activity of Oxidising Agents by Means of, 233
——, Uranium, Reaction of, 180
Salvarsan, Detection of, 153
————, the Detection of, New Method for, 452
" Salvarsan," Titration of, with Iodine Solution, 350
Sampling of Coal in the Mine, 527
Sand, Monazite, from Travancore, India, 523
Sandal-Wood Oil, African, Adulterant of, 146
Sand's Apparatus, Electrolytic Estimating of Copper and Zinc, Using, 182
Sands, Monazite, Analysis of, 522
——, Rare Earth-Containing (" Amang ") from Malay States, 523
Santolina chamæcyparissus, Essential Oil of, 589
Saponification and Acid Values in Wax, Estimation of, Simplified Process for, 355
——————, the Glycerol Method of, Effect of, upon the Fatty Acids, 425
—————— Value of Oils, Fats, and Waxes, Estimation of. Use of Propyl Alcohol, 290
Sauerkraut, Composition of, 548
(Scandium, Separation of Thorium from). Detection and Estimation of Thorium by Means of Iodic Acid, 436
Schaeffer's Curd Tester, Control Tests of the Colour Tests Used with, 378
Schardinger's Reagent, Behaviour of, towards Colostrum, 221
Schinus molle (L.), Essential Oil of, 68
Schloesing's Method, Gasometric Estimation of Nitrate by, and of Nitrite by Piccini's Method, Some Sources of Error in, 525
Schweinfurt Green, Arsenic in, Estimation of, 363
Scrap, Coke Crucible, Estimation of Carborundum in, 31
Sealed Tube Reactions, Gas in, Detection of, 531
Seeds of Leguminous Plants Grown in Togo and German East Africa, Composition of, 345
—— of *Lophira alata* (Banks), the Fat from, Composition of, 493
—— of the Ash-Tree, Oil from, 357
—— of *Xanthophyllum lanceolatum*, Oil from, 406
Sensitive Indicator, 26
———— Test for Phenol and Salicylic Acid, 295
Sensitiveness of the Colorimetric Estimation of Titanium, 310
—————— Tests by the Impact Machine (Drop Hammer), 314
Separating Funnel, Modified, and Washer, for Heavy Liquids, 89
Serum, Egg, and Milk Albumins, Coagulating-Points of, and of Serum Globulin, in Ammonium Sulphate Solutions, 416
———-Precipitin and the So-called Erythro-Precipitin, the Biological Reactions with, for the Detection of Blood, Comparative Values of, 590
Sewage Effluents and Water, the Amount of Nitrite in, the Determination of, and of the Amount of Dissolved Oxygen Absorbed by Sewage Effluents Containing Nitrites, 393
Seychelles Cinnamon Bark, 217
Shellac and Shellac Varnish, Analysis of, 464
Shrewsbury and Knapp's Process for the Estimation of Cocoanut Oil, Examination of, 195
Silicate Rocks, Ferrous Iron in, Determination of, 602
Silicates, Decomposition of, by Fusion with Boric Acid as Applied to Qualitative Analysis, 371
——, Ferrous Oxide in, the Titration of, by the Pebal-Dölter Method, Note on, 372
[illegible] Acid, Hydrated, in Clay, Determination of, 85

Silicon Alloys, Dissolving, 178
———, Ferrotitanium Alloys Containing Much, Analysis of, 373
———, in Vanadium and Molybdenum, and in their Iron Alloys, Estimation of, 311
Silicotungstic Acid, Estimation of Aconitine by, 142
Silver and Cadmium, Rapid Determination of, by Means of the Gauze Cathode and Stationary Anode, 473
—— and Gold, in Ores Containing Much Copper, Pot Assay of, 240
—— Anode and Mercury Cathode, Electrolytic Determination of Chlorine in Hydrochloric Acid with the Use of, 33
——, Assay of, by Means of the Touchstone, 372
——, Estimation of, by Electrolysis of an Ammoniacal Solution of its Oxalate, 308
——, German, and Other Alloys, the Separation of Nickel and Zinc in, Method for, 524
——, Iodine, and Alkali, Volumetric Solutions of, Use of Sulphur Dioxide in Checking Strengths of, 248
—— Vanadate, Precipitation of Vanadic Acid as, and Estimation of Phosphoric and Vanadic Acids in Presence of Each Other, 35
Simon and Lohrisch's Method, Estimation of Cellulose by, 77
Sizing, Animal, in Paper, Detection of. Differentiation of Gelatin and Casein, 290
Slag, Basic, Phosphoric Oxide in, Citrate-Solubility of, 32
Smokeless Powder, Camphor in, Estimation of, 420
————————, Stability Tests of, 295
Soap, in Heavy Grease, Estimation of, 559
——, Total Fatty Acids in, Estimation of, 20
Soaps, Different, Germicidal and Insecticidal Values of, with or without Associated Active Agents, 505
——, Fat, and Oils, the Total Fatty Acids in, Estimation of, 115
——, Fats, and Oils, Train Oil in, Detection of, 359
——, Fatty Acids in, Estimation of, 355
—— of the Alkalis and Alkaline Earths, Estimation of Free Fatty Acids in the Presence of, 595
SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS, PROCEEDINGS OF, 2, 43, 95, 132, 195, 263, 327, 387, 445, 489, 535, 577
Soda-Water, 16
Sodium Carbonate and Trimethylphenylammonium Carbonate, Precipitation of Zinc, Manganese, Cobalt, Nickel, Copper, and Cadmium, from Ammoniacal Solutions by Means of, 599
———, Micro-Chemical Test for, 372
——— Nitroprusside, Colour Reaction of Proteins with, 235
——— Paratungstate, in the Estimation of Carbon Dioxide in Carbonates and Nitrogen Pentoxide in Nitrates by Loss on Ignition, Application of, 526
——— Thiosulphate as Standard in Alkalimetry, 435
————————, Notes on the Formation of Hypoiodites and their Action on—A Source of Error in Certain Iodine Titrations, 132
Soil, Bacteriological Investigation of, 219
—— Phosphates, Solubility of, Effect of Ignition on, 371
Soils, Good and Poor, Dihydroxystearic Acid in, 504
——, Humus in, Estimation of, 597
——, the Electrical Conductivity of, Determination of, 183
——, the Examination of, Application of the Osmotic Pressure and Electrical Conductivity in, 184
Solid Hydride of Antimony, Attempts to Prepare, 300
—————— of Arsenic, Detection of, 300
Solids and Liquids, Vapour Pressures of, Static Method for Determining, 36

Soluble and Volatile Fatty Acids, Loss of, during the Ageing of Butter, 64
Solvents, Specifically Lighter, Automatic Extraction of Aqueous Liquids by Means of, 87
Soot, the Composition of, Note on, 120
Soxhlet Condenser, Improved, 528
Soya Bean Oil, 358
————, Composition of, 513
Spanish Marjoram (*Thymus mastichina*, L.), Essential Oil of, 69
——— Oil of Turpentine, 589
Sparteine, Reaction of, 559
Specific Gravities of their Components, Differences in, Quantitative Analysis of Mixtures by, 306
——— Gravity of Oils, Estimation of, 557
————— of Very Small Quantities of Milk, Determination of, 500
Specifications, Purchase of Coal under, 229
Spectroscopic Differentiation between Oxyhæmoglobin and Carbon Monoxide Hæmoglobin, and Hæmochromogen, 550
———— Test for Hæmoglobin in Urine, the Failure of, Hippuric Acid as a Cause of, 417
Spermaceti, Beeswax, Paraffin, etc., Fat in, Detection of, 496
Spirit, the Substitution of, for Aged Whisky, Detection of, 546
Spirits, Potable, the Analysis of, and its Bearing on the Definition of Whisky (Report), 441
———, the Analysis of, Colour Reactions of Aromatic Aldehydes and their Applicability in, 273
Spiritus Ætheris Nitrosi, Simple Volumetric Method for Testing, 454
Spongy Nickel, Preparation of, and the Precipitation of Nickel Compounds, 539
Stability (General) of Explosives in Various Countries, Methods of Testing, 79
——— Tests of Smokeless Powder, 295
Standard for Permanganate Solutions, Ferric Sulphate as, 520
——— in Alkalimetry, Sodium Thiosulphate as, 435
Standards in Acidimetry, Acids Suitable for Use as, 25
————, Portuguese, for Wines, Vinegar, and Olive Oil. (Extract from the Official Methods of Analysis), 502
Stannous Chloride, Estimation of Tungsten by Means of, 310
Starch and Glycogen, the Separation of, Method for, 501
———, Estimation of, 588
——— in Brewers' Grains, Estimation of, 500
———, Indigotin in the Presence of, Estimation of, 290
——— -Iodine Reaction, 28
——— Syrup, Commercial, the So-called Gallisin (Glucosin) Occurring in, Composition of, 543
————— in Jams, etc., Estimation of, 280
Static Method for Determining Vapour Pressures of Solids and Liquids, 36
Stearic and Palmitic Acids, Separation of Oleic Acid from, 23
Steel and Iron, Sulphur in, Volumetric Estimation of, 374
Vanadium in, Colorimetric Method for, 473
-, Carbon in, Estimation of, by Means of a Calorimetric Bomb, 562
-, Chrome Tungsten, Estimation of Chromium in, 83
-, Chromium in, Detection of, 428
-, Determination of Nickel in, Comparison of Methods for, 524
-, Nickel in, Rapid Estimation of, 373
Steels, Vanadium and Chrome-Vanadium, Manganese in, Estimation of, 604
——, ————————, Vanadium in, Determination of, 475

Sterilisation of Drinking Water by Means of Ultra-Violet Rays, 457
Stoppers, Rubber, Substitute for, 610
Stormer Viscosimeter, Consistency of Paints by the, 609
(Strontium and Calcium, Precipitant of), Racemic Acid as a Reagent, 365
————————, Sulphite Method for Separating and Identifying, 301
————, Barium, and Calcium, Quantitative Separation of, 364
————, Separation of, from Calcium, 247
Strychnine, Colorimetric Estimation of, 144
————, the Colour Reactions of, Influence of Certain Compounds upon, 296
Sublimed Sulphur, in a Mixture of Different Kinds of Sulphur, Estimation of, 374
Substitutes, and Cocaine, Identification of, 405
————, Turpentine, the Question of, a Further Contribution to, 207
Sucrose, Estimation of, and the Detection of Dextrin in Foods, 588
———, in Beet Sugar Factory Products, Determination of, by Clerget's Process, Using Invertase as Hydrolyst, 120
———, in the Presence of Other Sugars, New Method of Estimating, 28
Suction Pump, Simple Laboratory, 532
Sugar and Syrups, Estimation of Ash in, by the Determination of Electrical Conductivity, 298
——— Beets, Absorption of Arsenic by, 549
——— -Cane Juice, Acids in, Determination of, 594
———, Creatine, and Creatinine, in Urine, Colorimetric Estimation of, 591
———, in Blood, Estimation of, 73
———, Invert, in Honey, Detection of, 586
——— Juices and Syrups, Preparation and Constitution of Caramel and its Estimation in, 273
———, Polarimetric Estimation of, by the Method of Jolles, 361
——— Titration Method, Bang's, Preparation of the Copper Solution Used in, 501
Sugars, and Glycyrrhizin, in Liquorice Powder and Extracts, Estimation of, 409
——— in Feeding-Stuffs, Estimation of, 453
———, Other, Sucrose in the Presence of, New Method of Estimating, 28
———, Raw, and Other Products, the Polarisation of, upon Quartz Wedge Saccharimeters, Use of Temperature Corrections in, 297
———, Reducing, Estimation of, 298
Sulphate, Ammonium, Estimation of Sulphur in Coal-Gas and of, 309
————, Ferric, as Standard for Permanganate Solutions, 520
Sulphate, Hydrazine, Action of, on Nitrites, and a Method for the Estimation of Nitrite Nitrogen, 468
————, the Exact Determination of, Phenomenon of Occlusion in Precipitates of Barium Sulphate, and its Relation to, 426
Sulphates and Sulphuric Acid, New Method of Estimating, 178
Sulphides in Alkali-Metal Cyanides, Estimation of, 436
Sulphite Method for Separating and Identifying Calcium and Strontium, 301
Sulphites in Beer, the Chemical Behaviour and Preservative Action of, 495
Sulphites, Sulphurous Acid, and Complex Compounds of Sulphurous Acid, the Antiseptic Properties of, Experiments on, 285
Sulpho-Salicylic Reagent, Estimation of Nitrates in Water by, 85
Sulphur and Carbon in High-Grade Alloys of Tungsten, Molybdenum, and Vanadium, with Iron, Estimation of, 31
———, and Phosphorus in Organic Substances, Estimation of, 81
———, Carbon, Hydrogen, Oxygen, and Nitrogen, a Substance Containing, Analysis of, 553
———, ———, ————, ———, Nitrogen, Chlorine, Bromine, and Iodine, a Substance Containing, Analysis of, 553

Sulphur, Different Kinds of, a Mixture of, Estimation of Sublimed Sulphur in, 374
———, Dioxide in Commercial Gelatins, 271
———, ———, Use of, in Checking Strengths of Volumetric Solutions of Iodine, Alkali, and Silver, 248
——— in Coal, Determination of, by Means of Jackson's Candle Turbidimeter, 519
———, Estimation of, by the Method of Carius, 296
——— in Coal-Gas, Estimation of, and of Ammonium Sulphate, 309
——— in Iron and Steel, Volumetric Estimation of, 374
——— in Organic Compounds, Estimation of, 80
——— Monochloride, Use of, in the Determination and Analysis of Rare Earth Minerals, 567
———, Total, in Coal, Estimation of, 375
———, ———, in Coal-Gas, the Estimation of, "Perhydrol" for, 309
Sulphuric Acid and Sulphates, New Method of Estimation, 178
————— and Zinc, Separation of, 607
Sulphurous Acid, Complex Compounds of, Sulphites, and Sulphurous Acid, the Antiseptic Properties of, Experiments on, 285
—————— in Glucose Syrup, Estimation of, 543
—————— in White Wines, 351
Surface Factors, the Use of, and Errors [illegible] Determining Sizes of Mineral Grains, 182
Sweet Apricot Kernels, Composition of, 450
Sweetened Condensed Milk, Analysis of, 138
Syrup, Commercial Starch, the So-called Gallisin (Glucosin) Occurring in, Composition of, 543
——, Glucose, Sulphurous Acid in, Estimation of, 543
——, Starch, in Jams, etc., Estimation of, 280
Syrups and Juices, Sugar, Preparation and Constitution of Caramel and its Estimation in, 273
——— and Sugar, Ash in, Estimation of, by the Determination of Electrical Conductivity, 298
Tan Liquors, the Estimation of the Acidity of, Electrometric Method for, 121; Part II., 155
Tannin Estimation, Nickel Hydroxide in, Use of, 516
——— in Solution, Detection of, 412
in Wines, Estimation of, 412
—, Quantitative Estimation of, by Means of Casein, 123
Tantalum and Columbium, Indirect Method for Determining, 239
— the Estimation of, 310
-, Separation of, 239
Tap-Funnel, Adaptation of, to Rapid Electro-Analysis with Stationary Electrodes, 313
Tar and Tar Constituents in Gas, New Method of Estimating, 299
—, Coal, the Residues from the Distillation of, Estimation of Rosin in, 463
—, Distillation of, Flask for, 529
— Oils and Creosote Available for Wood Preservation, Characters of, 228
Tarry Substances in Dark Mineral Cylinder Oils, Estimation of, 298
Tars, Coal, and Refined Tars, Oils, and Pitches Derived Therefrom, Methods for Testing, 298
—— Containing Water, Distillation of, 171
Tartaric Acid, Estimation of, 148
—————, in Apples, Pears, Cider, and Perry, Estimation of, 545
—————, Total, Estimation of, 148
—————, ——, —————, by Goldenberg's Method, 81
Tea, Valuation of, 454
Tellurium, Estimation of, 179

Water and Sewage Effluents, the Amount of Nitrite in, the Determination of, and of, the Amount of Dissolved Oxygen Absorbed by Sewage Effluents Containing Nitrites, 393
——, Bacteriology of: Its Present Position, 285
——, Drinking, Sterilisation of, by Means of Ultra-Violet Rays, 457
——, Estimation of Nitric Oxide, and its Behaviour towards, 433
—— Gas Calorimeter, Still, 250
—— in Honey, Estimation of, 586
——, Nitrates and Nitrites in, Detection of, 432
——, Nitric Acid and Nitrous Acid in, Detection and Estimation of, 433
——, Nitrites in, Detection of, 370
——, Nitrous Acid, and Nitric Acid in, Detection and Estimation of, 433
——, Organic Substances in, Estimation of, by Means of Permanganate, 502
——, River, Typhoid Bacilli in, Vitality of, with Special Reference to the Question of Storage, 185
——, Sea, the Salinity of, Estimation of, by Means of the Refractometer, 311
——, the Estimation of Nitrates in, the Phenol-sulphonic Acid Method for, Study of. III. Chief Sources of Error, 244
——, ————, the Phenol-sulphonic Acid Method for, Study of. IV. A Modified Method, 244
——, Small Quantities of, in Lard, Estimation of, 497
——, the Hardness of, Estimation of: The Wartha-Pfeiffer Method and its Modification, 311
——, Traces of, in Ether, Method for the Accurate Estimation of, 420
Waters, Mineral, Arsenic and Iron Salts in, Estimation of, 466
Wax, Acid and Saponification Values in, Simplified Process for Estimation of, 355
—— and Fatty Oil Obtained from Coffee Berries, 106
——, Candelilla, 598
Waxes, Fats, and Oils, the Saponification Value of, Estimation of. Use of Propyl Alcohol, 290
Wendler's Method for the Estimation of Fat in Milk, 14
Wet Method for the Estimation of Carbon in Organic Substances, 354
Wheat and Rye Bran, Customs Examination of, 350
—— Gluten, Influence of Certain Vegetable Proteins on, 407
Wheaten Flour, the Quality of, Method of Estimating, 543
Whisky, Aged, the Substitution of Spirit for, Detection of, 546
White Metals, the Analysis of, Rapid and Accurate Method for, 606
—— Wines, Sulphurous Acid in, 351
Wine, Invertase in, Detection of, 350
——, the Volatile Acids in, Estimation of, 502
Wines, British, the Composition of, Note on, 60
——, Tannin in, Estimation of, 412
——, Vinegar, and Olive Oil, Portuguese Standards for. (Extract from the Official Methods of Analysis), 502
——, White, Sulphurous Acid in, 351
Wiping, Purifying and Drying Organic Liquids by, Method for, 26
Wolfram Concentrate, the Assay of, 398
Wood-Fungi and Cereals, Pentosans and Methylpentosans in, Estimation of, 460
————, Certain, Pentosans of, 162
Wood Preservation, Creosote and Tar Oils Available for, Characters of, 228
—— Turpentine Oil, Turpentine Oil, and Rosin Essence, Reaction of, 107
Wool-Grease Oleine, Mineral Oil in, Detection of, 29
———— Oleines, Examination of, 29
————, French, Rosin Oils in, Detection of, 30
Wool Oils, Unsaponifiable Matter in, Estimation of, 168

Xanthophyllum lanceolatum, Oil from the Seeds of, 406

Yeast and Beef Extracts of Known Origin, Comparison of, 64
—— and Meat Extracts of Known Origin, Comparison of, 104
Yellow Pigmentary Substances, Normal, of Urine, Relation of, to the Diazo-Reaction, and a Colorimetric Method for Estimating Urochrome and Urochromogen, 223

Zinc and Copper, Electrolytic Estimation of, Using Sand's Apparatus, 182
—— and Sulphuric Acid, Separation of, 607
—— and Nickel in German Silver and Other Alloys, the Separation of, Method for, 524
——, Manganese, Cobalt, Nickel, Copper, and Cadmium, Precipitation of, fro Ammoniacal Solutions by Means of Sodium Carbonate and Trimethy phenylammonium Carbonate, 599
——, the Volumetric Estimation of, Reagent Paper for, 126
——, Volumetric Estimation of Ferric Iron by Permanganate after Reduction with, 521
Zygadenus Intermedius ("Death Camas"), Analysis of, 156

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THE ANALYST.

INTERNATIONAL ATOMIC WEIGHTS, 1911.

		O=16.			O=16.
Aluminium	Al	27·1	Molybdenum	Mo	96·0
Antimony	Sb	120·2	Neodymium	Nd	144·3
Argon	A	39·88	Neon	Ne	20·2
Arsenic	As	74·96	Nickel	Ni	58·68
Barium	Ba	137·37	Nitrogen	N	14·01
Bismuth	Bi	208·0	Osmium	Os	190·9
Boron	B	11·0	Oxygen	O	16·00
Bromine	Br	79·92	Palladium	Pd	106·7
Cadmium	Cd	112·40	Phosphorus	P	31·04
Cæsium	Cs	132·81	Platinum	Pt	195·2
Calcium	Ca	40·09	Potassium	K	39·10
Carbon	C	12·00	Praseodymium	Pr	140·6
Cerium	Ce	140·25	Radium	Ra	226·4
Chlorine	Cl	35·46	Rhodium	Rh	102·9
Chromium	Cr	52·0	Rubidium	Rb	85·45
Cobalt	Co	58·97	Ruthenium	Ru	101·7
Columbium	Cb	93·5	Samarium	Sa	150·4
Copper	Cu	63·57	Scandium	Sc	44·1
Dysprosium	Dy	162·5	Selenium	Se	79·2
Erbium	Er	167·4	Silicon	Si	28·3
Europium	Eu	152·0	Silver	Ag	107·88
Fluorine	F	19·0	Sodium	Na	23·00
Gadolinium	Gd	157·3	Strontium	Sr	87·63
Gallium	Ga	69·9	Sulphur	S	32·07
Germanium	Ge	72·5	Tantalum	Ta	181·0
Glucinum	Gl	9·1	Tellurium	Te	127·5
Gold	Au	197·2	Terbium	Tb	159·2
Helium	He	3·99	Thallium	Tl	204·0
Hydrogen	H	100·8	Thorium	Th	232·0
Indium	Id	114·8	Thulium	Tm	168·5
Iodine	I	126·92	Tin	Sn	119·0
Iridium	Ir	193·1	Titanium	Ti	48·1
Iron	Fe	55·85	Tungsten	W.	184·0
Krypton	Kr	82·9	Uranium	U	238·5
Lanthanum	La	139·0	Vanadium	V	51·06
Lead	Pb	207·10	Xenon	Xe	130·2
Lithium	Li	6·94	Ytterbium (Neoytterbium)	Yb	172·0
Lutecium	Lu	174·0	Yttrium	Y	89·0
Magnesium	Mg	24·32	Zinc	Zn	65·37
Manganese	Mn	54·93	Zirconium	Zr	90·6
Mercury	Hg	200·0			

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE ordinary monthly meeting of the Society was held on Wednesday evening, December 7, in the Chemical Society's Rooms, Burlington House. In the absence of the President, the chair was occupied by Mr. A. E. Ekins, F.I.C., Vice-President.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. C. L. L. Claremont, B.Sc., A.I.C., and H. D. Elkington, were read for the second time; and certificates in favour of Messrs. Ronald Leslie Collett, B.A. (Cantab.), A.I.C., Little Dawley, Hayes, Middlesex, assistant to Mr. A. Chaston Chapman, F.I.C.; William Beaverley Cowie, 26, Clyde Street, Edinburgh; Frank Evershed, analyst and consulting chemist to Messrs. Clarke, Nicholls and Coombs, Ltd., Victoria Park, London, N.E.; and George Taylor, 6, Vicarage Park, Plumstead, S.E., assistant to Dr. Bernard Dyer, F.I.C., were read for the first time.

Messrs. G. Jörgensen and F. F. Shelley, F.I.C., were elected members of the Society.

The following papers were read: "On Fischer's Modification of Volhard's Method for the Estimation of Manganese, and its Comparison with Other Well-known Methods," by Edward Cahen, A.R.C.Sc., F.I.C., and Harry F. V. Little, A.R.C.Sc., B.Sc.; "A New Volumetric Process for the Estimation of Tungsten," by E. Knecht, Ph.D., F.I.C., and Miss E. Hibbert; "A New Volumetric Process for the Estimation of Molybdenum," by E. Knecht, Ph.D., F.I.C., and F. W. Atack; "Note on the Composition of British Wines," by Edward Russell, B.Sc., F.I.C., and T. R. Hodgson, M.A., F.I.C.; "The Degree of Accuracy with which the Proteins of Milk can be Estimated by the Aldehyde Method," by H. Droop Richmond, F.I.C.; "Note on Gorgonzola Cheese," by Edward Hinks, B.Sc., F.I.C.; "Tests for Cocaine and Certain Cocaine Substitutes," by E. H. Hankin, M.A., Sc.D.; and "Some Analyses of *Hevea* Latex," by Clayton Beadle and Henry P. Stevens, M.A., Ph.D., F.I.C.

* * * * *

TESTS FOR COCAINE AND CERTAIN OTHER ANÆSTHETICS.

BY E. H. HANKIN, M.A., Sc.D.

(*Read at the Meeting, December* 7, 1910.)

COCAINE is a substance the tests for which do not work so well in practice as might be expected from textbook statements. Even the permanganate test, which is stated to be delicate, should, according to Autenreith ("Detection of Poisons," translated by Warren, p. 85), be carried out with a concentrated aqueous solution of a cocaine salt. I have discovered some modifications of this test, by means of which extremely minute quantities of cocaine can be readily detected, and also clearly distinguished from tropacocaine and other allied cocaine substitutes.

If a drop of a solution of cocaine and a drop of a solution of permanganate of potassium are placed together on a glass slide, it frequently happens that on microscopical examination no crystals of cocaine permanganate are seen, but only a dark red viscous mass. If the two drops, however, are rubbed together at the moment of mixing with a glass rod, so that the red coagulum is pressed and squeezed, there is a much greater chance of the characteristic crystals of cocaine permanganate being formed. I noticed also that the formation of the crystals was hindered by any condition that tended to dilute the cocaine. To prevent this effect, I tried precipitating the cocaine by the addition of ammonia before the addition of the permanganate. This I found to be advantageous, especially when dealing with minute quantities. The delicacy of the test was found to be increased by the substitution of bromine-water for ammonia. A trace of cocaine is placed on a glass slide in a minute drop of a one-third or one-quarter saturated solution of bromine in water. A drop of an acid solution of permanganate (4 per cent. potassium permanganate and 3 per cent. sulphuric acid freshly mixed) is added. If the drops are mixed thoroughly, the formation of crystals is generally immediate. If the drops are not thoroughly mixed, the cocaine permanganate crystals are usually formed more slowly, and may take the form of large branched aggregates that give a remarkable play of colour with polarised light.

In a case of cocaine smuggling, I received for examination the ashes of a fire in which cocaine was suspected to have been burnt. Embedded in the ashes was a piece of paper about an inch square that was charred only along one edge. Small pieces of this paper, only a few millimetres square, treated with sufficient bromine-water to wet them, and then placed on a glass slide with the acid permanganate solution, in four cases out of five gave typical crystals of cocaine permanganate. I also obtained permanganate of cocaine crystals from the Stas-Otto alkaline ether extract of a portion of the ashes.

Though the test as thus carried out is extremely delicate, it cannot be regarded as reliable. Some unknown factors appear to come into play, perhaps connected with the relative amounts of the different reagents, which may in certain cases hinder or prevent the formation of the crystals.

While trying the action of permanganate on certain cocaine substitutes, I discovered an improved method of carrying out the test. I found that in the case of scopolamine, with a dilute solution of permanganate, oily red drops were produced; while if a stronger solution of permanganate was employed, crystals of permanganate of scopolamine were frequently formed. This clue led me to a better method of applying the permanganate. A drop of a strong solution of permanganate was spread out on a glass slide and allowed to dry. The "permanganate film" thus produced was found to be of use in testing, not only for cocaine, but also for certain cocaine substitutes. As will be described below, if a drop of a solution of either alypin, tropacocaine, or scopolamine, is placed on such a permanganate film, covered with a coverslip, and examined with the microscope, characteristic crystals of the permanganate compounds will, as a rule, be seen. On applying this method to cocaine, I found that generally no crystals, but only oily red drops, were produced. It occurred to me to attempt to delay or modify the action of the permanganate by the addition of some indifferent salt. I found that a saturated or half-saturated solution of alum was suitable for the purpose. If a trace of cocaine is dissolved in a saturated solution of alum, and a small drop of this solution is placed on a permanganate film, and covered with a coverslip (which should be dropped into position, and not rubbed about), then, within a minute or two, numerous crystals of cocaine permanganate are formed. This test is extremely delicate, reliable, and easy to carry out. I have had the following opportunities of using it practically:

In a case of illicit dealing in cocaine, the culprit appears to have received information beforehand that his shop was to be raided. Consequently the police found no cocaine. But, by the alum method above described, cocaine was detected by me on ten out of eleven pieces of paper that had been found, and that were supposed by the police to have been used in wrapping up doses of cocaine. In carrying out the test, from the centre of each piece of paper I cut out as small a fragment as could readily be handled with a forceps. This fragment was placed for a minute in a small drop of alum solution, then removed, and the drop of alum solution was transferred to a permanganate film. On covering with a coverslip and examining under the microscope, numerous cocaine permanganate crystals were seen to develop. After carrying out the test, I asked my assistant, Dr. Gore, to repeat my observation. Although his acquaintance with this test was confined to my description, he succeeded in obtaining permanganate crystals from each of the eleven pieces of paper. In another case a telegraph form was sent for examination. On this was a stain, supposed to be of saliva, of a person who had taken cocaine. Scrapings of this stain suspended in alum solution gave the cocaine permanganate reaction.

On applying the permanganate test to various cocaine substitutes, I obtained results that may be summarised as follows:

1. Cocaine and alypin form crystals that often have the form of delicate pink plates.
2. Tropacocaine easily forms crystals that usually have a red or dark red colour.
3. Scopolamine forms with difficulty crystals of red colour.
4. Beta-eucaine and stovaine form reddish oily drops, but no crystals.
5. Novocaine, holocaine, and nirvanine, turn brown instantaneously, with formation of finely granular brown precipitate.

In each of the above four cases in which crystals are formed, these may occur as isolated plates or prisms, or as rosettes, or as larger aggregates. But the crystals differ both in appearance and in the conditions of their formation. The following are details of my observations:

1. *Cocaine.*—As already stated, cocaine permanganate crystals are best formed on a permanganate film, the cocaine having been dissolved in a saturated solution of alum. They are less readily formed, or may not appear at all, if the cocaine is dissolved in water. They are still less likely to develop if the cocaine is dissolved in a strong solution of potassium bromide. Single cocaine permanganate crystals are nearly square in shape. Larger aggregates of these crystals usually have right-angled notches or indentations. In testing for lactate of cocaine, it is advisable to treat its solution with ammonia and shake up with ether. The ether is placed in a

separating funnel and shaken with a small quantity of a solution of alum. The cocaine passes into the alum solution, which may then be separated and tested with a permanganate film.

2. *Alypin.*—These crystals are best formed on a permanganate film. They are most easily obtained if the alypin is dissolved in water. If the alypin is dissolved in a strong solution of potassium bromide, the crystals form readily (differing thus from cocaine), and often assume the form of isolated plates or small rosettes. When alypin is used dissolved in water, the crystals usually take the form of irregular branched aggregates or narrow and dented plates and spikes. Alypin does not readily form permanganate crystals when dissolved in a solution of alum.

3. *Tropacocaine.*—Tropacocaine forms crystals with permanganate more readily and under a greater variety of conditions than any other cocaine substitute. If a minute quantity of tropacocaine is dissolved in a saturated solution of alum and placed on a permanganate film, crystal aggregates are formed of reddish-violet colour, and which, being usually curved, have a feathery or fernlike appearance. If a drop of a dilute aqueous solution of tropacocaine is mixed with a drop of a dilute solution of permanganate, isolated crystals or smaller crystalline aggregates are deposited. These are of varying or irregular shape. In some cases they are of oblong outline, with a slight curvature difficult to see along one side.

4. *Scopolamine.*—Scopolamine permanganate is less easy to obtain in the crystalline form than any of the previously described substances. It is necessary to use a strong aqueous solution and a permanganate film. With more dilute solutions only a granular precipitate or oily drops are, as a rule, obtained. The crystals are dark red in colour usually, and of very variable form. Sometimes they occur as prisms pointed at both ends, thus having an irregular hexagonal outline, or the prisms are pointed at one end and blunt at the other. Smaller crystals may be of irregular oblong shape. Larger aggregates are not often seen.

5. *Saporetti's Bromine Test applied to Other Cocaine Substitutes.*—Saporetti's observation of the effect of bromine-water on cocaine substitutes furnishes a test applicable in those cases in which permanganate is not of use. "Intorno ad una nuova reazione distintiva fra eucaina-alpha e beta. Differenziazone fra cocaina e suoi succedanei" (*Bolletino Chemico-Farmaceutico*, July, 1909, p. 479).

According to Saporetti, bromine-water has the following actions, which accord with my own observations:

1. With cocaine, scopolamine, stovaine, and novocaine, a yellow precipitate is produced, soluble on warming.

2. With beta-eucaine, a yellow precipitate partly soluble on warming, but which comes down again as a white precipitate on boiling.

3. With nirvanine, a yellow precipitate soluble on warming. The liquid then acquires a reddish colour and emits a pleasant smell.

4. With holocaine (not mentioned by Saporetti), I find that bromine-water produces a yellow precipitate. On boiling, the yellow precipitate is dissolved, and the liquid acquires a pinkish-white opalescence.

Conclusions.—1. The permanganate test for cocaine is of great delicacy if, as above described, the cocaine is dissolved in alum solution and the permanganate is used in the form of a dried film.

2. The test forms a ready means of distinguishing between cocaine and certain cocaine substitutes.

3. Of these substitutes, alypin, tropacocaine, and scopolamine, form crystalline permanganates, which can be readily distinguished from each other and from the permanganate of cocaine.

4. Beta-eucaine, stovaine, novocaine, holocaine, and nirvanine, form no crystals with permanganate. Saporetti's bromine-water test furnishes a means of distinguishing these substances.

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SOME ANALYSES OF HEVEA LATEX.

BY CLAYTON BEADLE AND HENRY P. STEVENS.

(*Read at the Meeting, December* 7, 1910.)

AMONG the few analyses of *Hevea Braziliensis* latex which have been published, we should like to refer to an analysis of Faraday's, as it is incorrectly quoted in Weber's "The Chemistry of India Rubber," from which it has been copied from one textbook to another, and has just appeared again with the same errors in Hinrichsen and Memmler's "Der Kautschuk und seine Prüfung." The analysis is invariably quoted as that of the latex of *Hevea Braziliensis*, while no statement is made as to its botanical origin in Faraday's original paper (*Quarterly J. of Science*, 1826, **21**, 19); we are only told that it came from Mexico. One of us has experimented with latices of various origins in the East, and we have little doubt but that the latex analysed by Faraday was derived from the *Castilloa elastica.* We base this opinion on the data given by Faraday, who found the sp. gr. to be greater than that of water, while the sp. gr. of *Hevea* latex is always less than unity. Faraday's description of the creaming of his latex, on which he based a method for removing the water-soluble constituents, is characteristic of *Castilloa* latex. It is the principle employed at the present day for the preparation of this class of rubber in Mexico. It is hardly necessary to labour this point any further, beyond mentioning, as further characteristics of *Castilloa* latex, the dark colour, bitter taste, acid reaction, and excellent state of preservation, all of which are noted by Faraday in the paper mentioned above.

The more recent analyses have been carried out in Europe on samples of latex to which a preservative had been added, and there is no guarantee that the latex was either (1) free from added water, or (2) an average sample.

As regards the first point, the common practice of putting a little water in the collecting cups, as well as the possibility of showers during tapping, makes it extremely likely that the analyses, so far published, show too high a percentage of water.

As to the second point, latex from trees to all appearance of similar size and girth, growing in the immediate neighbourhood of one another, give results, on analysis, showing considerable variation in percentage of dry solids. It is therefore necessary to carefully supervise the collection of latex, and to work on the average latex from a large number of trees.

The determinations made consisted of total dry solids, total caoutchouc, and the ordinary commercial analysis of the latex, including the figures for acetone extract, nitrogen, and ash. For the total solids, a weighed quantity of the latex (about 10 c.c.) was evaporated at about 80° C., and then dried in a steam-oven at 100° C. till it ceased to lose weight. The total caoutchouc was obtained by pouring a weighed quantity of latex (about 10 c.c.), in a thin stream, into 50 c.c. of alcohol. After standing, the coagulum was removed, pressed, and dried at 100° C. For further analysis, the total solids obtained as described were put into a small stoppered bottle with well-fitting stopper, and sent home. The acetone extract (resin), the protein (N × 6·25),* and the ash, were estimated in the usual manner.

The percentage of total solids and caoutchouc depends on the manner and frequency of tapping. For trees of an average age of seven or eight years, lightly tapped every other day, the total dry solids were found to be approximately 40 per cent. The sp. gr. varied from 0·980 to 0·972. In another series of experiments where the same trees were more heavily tapped, the total dry solids were approximately 30 per cent. The total solids in the latex, other than caoutchouc, amount to about 2½ per cent. on the latex, so that by subtracting 2·5 from the figure for total dry solids, we obtain the approximate figure for caoutchouc. Of the samples examined, the highest figure for caoutchouc was 43 per cent., and the lowest 18·5 per cent. In all cases the latex analysed was the mixed product of at least one hundred trees. The figures are taken from results obtained when tapping in dry weather only, and in no case was any water put into the cups or added to the latex.

As to the influence, if any, of the age of the tree on the composition of the latex, it is well known to all rubber planters that when tapped for the first time the young *Hevea* tree yields very little latex during the first few days, the amount gradually increasing until, after a time, it remains constant round about a certain figure. Determinations carried out on trees about four years of age, and just brought into tapping, yielded in one series of experiments 22 to 25 per cent., and in another 27 to 33 per cent. of dry rubber in the latex. The trees were tapped every day and on different systems, but not heavily. The results are the average figures over periods of some months. The figures, taken in conjunction with others, where the trees were older, but tapped under similar conditions, show that the latex from very young trees is poorer in caoutchouc than that from older trees.

An examination was made of latex from the primary tissue of the leaves and stems of leaves. If the stem of a leaf be snapped in two, a drop of latex exudes from the exposed tissue. This rapidly coagulates, owing to the abundant acid juices in the sap vessels, which are broken at the same time. The tiny clot of rubber can be picked off with a small pair of tweezers. From several hundred leaves sufficient of the coagulated latex was obtained for analysis. Owing to the rapid coagulation just referred to, the amount of moisture could not be determined; but the mass was dried and analysed in the ordinary manner. The dried latex is at first rather tacky; but on keeping a month or two, and passing once or twice between steel rolls, it is no longer adhesive, and somewhat resembles a high-class purified guayule rubber. It is pale in colour, but rather soft, and much weaker than the rubber from the bark of mature trees. The analyses of this material, together with those of dried latex from four- and ten-year-old trees, are given below.

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* This factor, as Spence has shown, is probably too high; but as the proteins of rubber have not yet been isolated, the above figure is adhered to. This course has also the advantage of allowing a comparison to be drawn between our analytical results and those of previous workers.

Analyses of the latex are also given for trees of these ages, taking the percentage of moisture 70 per cent. and 60 per cent. respectively, these being the figures arrived at for average results when trees are tapped on a conservative basis. The dried latex contained about 1 per cent. of moisture, but in the analyses the figures are given as calculated for dry material.

	Dried Latex from Stems of Leaves.	Dried Latex from Trees Four Years Old.	Dried Latex from Trees Ten Years Old.
	Per Cent.	Per Cent.	Per Cent.
Acetone extract (resin) ...	7·12	4·06	4·13
Protein	13·02	4·90	5·08
Ash	1·19	0·80	1·75
Caoutchouc (by difference) ...	78·67	90·24	89·04

COMPOSITION OF LATEX.

	Trees Four Years Old. Per Cent.	Trees Ten Years Old. Per Cent.
Water...	70·00	60·00
Acetone extract (resin) ...	1·22	1·65
Protein	1·47	2·03
Ash	0·24	0·70
Caoutchouc (by difference)...	27·07	35·62

We have also examined the aqueous extracts from the total solids of the above latices in respect of their reducing action on Fehling's solution, and have obtained the following results calculated in terms of glucose on the rubber:

	Before Inversion.	After Inversion.
	Per Cent.	Per Cent.
Latex from ten-year-old trees	None	0·34
„ „ four „ „	Trace	0·79
„ „ the petioles or leaf-bases	0·36	2·25

These figures are interesting, as pointing to the much larger percentages of carbohydrates contained in the latex from primary tissue; they are, however, only of relative value, and do not show the actual amounts of sugars in the original latices, as these are partially destroyed by evaporation of the latex to dryness.

The appearance under the microscope of the latex from the petioles of leaves is interesting and suggestive. A relatively small number of caoutchouc globules are to be seen of the ordinary appearance. These stand out brightly, on a groundwork composed of a much larger number of smaller globules of about one-third of the average size of the caoutchouc globules. These small globules are in rapid Brownian movement, and are not so well defined as the caoutchouc globules, having, apparently, a much lower refractive index.

It is interesting to note that the solids contained in the latex from the petioles differ from that from the bark, chiefly in the much higher content of nitrogenous matter. There is also nearly another 3 per cent. of resins or other matter soluble in acetone. The acetone extracted rubber from the latex of the petioles, when allowed to stand for a few months, is fairly tough and elastic, and resembles rubber from the bark of mature trees similarly treated.

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THE DEGREE OF ACCURACY WITH WHICH PROTEINS CAN BE ESTIMATED IN MILK BY ALDEHYDE TITRATION.

BY H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, December* 7, 1910.)

IN a paper on the aldehyde figure of milk, Steinegger (*Zeitsch. Untersuch f. Nahr. Genussm.*, 1905, **9**, 659) gave some determinations showing that this figure was approximately proportional to the total nitrogen. Miller and I (ANALYST, 1906, **31**, 224) modified Steinegger's procedure chiefly by substituting strontia for soda solution as the alkali used for titration, and under our conditions we found a higher "aldehyde figure" than that yielded by soda titration, the ratio being approximately 1·1, but not being constant; we attributed this to the degree of dissociation of the sodium and strontium salts differing.

While the correctness of this view is strengthened by the conclusions of J. B. Robertson (*J. Phys. Chem.*, 1910, **14**, 601), I find that there is another disturbing factor. Formaldehyde solution is almost invariably acid, and when the strong solution is neutralised to phenolphthalein with soda, and then diluted with neutral water, it becomes appreciably alkaline; while, if neutralised with strontia, the reaction is unaltered by dilution. This fact alone would cause the soda aldehyde figure to be lower, to an extent varying with the acidity of the formaldehyde solution, than the strontia figure. I have also found that the amount of formaldehyde recommended by Steinegger (*loc. cit.*) as developing the maximum acidity (6 per cent. of 40 per cent. formaldehyde solution) is not quite sufficient, as the addition of larger quantities results in the development of slightly more acidity.

From the experiments of Miller and myself (*loc. cit.*), I deduced the factor 0·171 for converting aldehyde figures (expressed as c.c. of $\frac{N}{1}$ alkali per litre of milk) into percentages of proteins. As we showed that there was a considerable difference between the aldehyde factors of casein and albumin, it is evident that if a protein estimation is to be calculated from the aldehyde figure, it must be assumed that the ratio of casein to albumin does not appreciably vary, and that other proteins must be present in negligibly small quantities. Among these other proteins must be included the products of hydrolysis due to the action of micro-organisms or of enzymes. Miller and I (ANALYST, 1906, **31**, 328; 1908, **33**, 115) have shown that as milk ages the aldehyde figure rises, doubtless from this cause.

Although we used more formaldehyde than the amount recommended by Steinegger, further experience has led me to believe that we may not in all cases, especially with high aldehyde figures, have employed enough, and again, in some cases we used milks which were of some age; these causes would lead to errors in opposite directions.

I have carried out a series of determinations following the method given below, and using only fresh milk, with the object of ascertaining the agreement between duplicate determinations and the constancy of the factor.

The method is: To 10 or 11 c.c. of milk at least 1 c.c. of 0·5 per cent. phenolphthaleïn solution is added, and the milk neutralised with $\frac{N}{11}$ (approximately) strontia solution; to the faintly pink liquid 2 c.c. or more of Schering's 40 per cent. formaldehyde solution is added, and the titration continued till the same degree of pink colour appears. After deducting the acidity of the formaldehyde solution, the latter titration represents the aldehyde figure. At the same time an estimation of nitrogen by the Kjeldahl method is made, and the proteins calculated by the factor 6·38.

In the experiments given all the aldehyde determinations are the mean of two, three, or, in one case, four titrations. The worst experiment was this: 20·2°, 19·8°, 20·2°, and 21·0°, a maximum difference of 1·2°; while the best consisted of three concordant figures. From forty-eight experiments the probable error of a single titration was calculated to be ± 0·2°.

The table below gives the experiments:

Aldehyde Figure C.C. $\frac{N}{1}$ Acid per Litre.	Proteins N × 6·38.	Ratio $\frac{P}{A}$.	Proteins Calculated.	Difference.
6·6	1·16	0·176	1·12	−0·04
10·1	1·74	0·172	1·72	−0·02
13·6	2·31	0·170	2·31	—
16·1	2·76	0·171	2·74	−0·02
16·8	2·88	0·171	2·86	−0·02
18·5	3·13	0·169	3·14	+0·01
19·0	3·22	0·1695	3·23	+0·01
19·5	3·38	0·173	3·32	−0·06
19·6	3·40	0·174	3·33	−0·07
19·7	3·38	0·172	3·35	−0·03
20·2	3·44	0·1705	3·43	−0·01
20·2	3·47	0·172	3·43	−0·04
20·3	3·44	0·1695	3·45	+0·01
21·4	3·73	0·174	3·64	−0·09
21·7	3·66	0·169	3·69	+0·03
24·0	3·95	0·165	4·08	+0·13
25·0	4·15	0·166	4·25	+0·10
23·7	4·54	0·192	4·03	−0·51

The last sample was of abnormal composition—

Sp. Gr.	Total Solids.	Fat.	Milk Sugar.	Proteins.	Ash.	Solids-not-Fat.	Acidity.
1·0283	13·20	5·00	2·87	4·54	0·89	8·20	12·0°

and I have excluded it from the figures used for calculating the mean factor, not only on this account, but because it would be excluded by the application of Chauvenet's criterion (*cf.* Mellor's "Higher Mathematics," p. 476). A roughly quantitative estimation showed that the nature of the proteins was abnormal.

From the remainder of the figures I calculate the factor 0·170 as the most probable figure to express the relation between proteins and aldehyde figure, which is in practical agreement with that formerly deduced. The maximum errors given in the table are +0·13 and −0·09, and the maximum errors of single determinations are +0·20 and −0·13.

In the experiments given above I have searched for milks which depart from the average as much as possible, and have tried to avoid mixed milks of average composition. I am therefore justified in concluding that the aldehyde determination gives a very rapid and approximately accurate determination of the proteins in milk, with the reservation that with rare abnormal samples the error is considerable. I consider that the method is of the same order of accuracy as the centrifugal methods for the estimation of fat, or the calculation method of deducing the solids-not-fat from fat and specific gravity.

DISCUSSION.

Mr. C. REVIS said that lately he had been making some comparative experiments on the estimation of casein, and had found the aldehyde method to be second in accuracy to the method of precipitation with magnesium sulphate. Personally he preferred soda to strontia for neutralising the acidity of the milk. The factor for calculation was different, but the final results were the same. The extraordinary results obtained in the case to which Mr. Richmond had drawn special attention were no doubt due, not to any fault in the method, but to abnormality of the sample. From its composition he should imagine that the cow had either been abnormally long in milk or was emaciated from disease. He should be inclined to think that the proteins in this case did not consist simply of casein and albumin, but included other proteins, the presence of which was due to disease.

Mr. L. M. NASH asked whether it would be possible to distinguish by means of the aldehyde figure between coagulable and non-coagulable proteins—that is to say, to determine the percentage of casein in presence of lact-albumin, or to determine the percentage of actual casein in commercial casein.

Mr. H. D. RICHMOND said that his reasons for preferring strontia to soda for aldehyde titration had been given in the paper; but, of course, soda could be used if a different factor were employed. In the case of the abnormal sample the suggestion of disease was not tenable, because the cows were regularly examined by a veterinary surgeon and reported healthy. Coagulable and non-coagulable proteins could not be distinguished by this method, because it was simply a method for estimating the acidity developed on the addition of formaldehyde to proteins, and, since both casein and albumin contained amino-acid groups, they both gave the reaction, though their aldehyde figures were not the same. He had, however, used the method for the approximate determination of the proteins in commercial casein, but, since the total volume of alkali used was only 3 or 4 c.c., the results, with a substance that might contain 70 per cent. of casein, could not be expected to be very accurate.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Analysis of Commercial Absinthe. L. Ronnet. (*Ann. Falsif.*, 1910, **3**, 477-480.)—The following process is recommended for the analysis of absinthe and other similar liqueurs, as, in addition to the estimation of the quantity of "essences" present, it affords a means of ascertaining whether brandy or commercial alcohol has been employed in the preparation of the liqueur. A volume of 250 c.c. of the sample is rinsed into a flask with 25 c.c. of water and distilled, 245 c.c. of distillate being collected. The distillate is then diluted with water to a volume of 250 c.c., and the alcoholic strength of the solution is determined (according to French law absinthe liqueur must not contain more than 65 per cent. of alcohol). The iodine absorption of the distillate is now determined, 50 c.c. of the distillate being treated with Hübl's solution for this purpose. As 1 grm. of the mixed oils constituting the "essences" present in absinthe absorbs 1·328 grms. of iodine, the quantity of "essences" in the sample may be calculated from the iodine absorption found (French law requires that not more than 3·5 grms. of total "essences" nor more than 1 grm. of wormwood oil shall be present per litre of liqueur). The remainder of the distillate is now diluted with water until it contains exactly 50 per cent. of alcohol, and 200 c.c. of the dilute alcoholic solution thus obtained are shaken in a separating funnel with about 50 c.c. of olive oil (by volume) which has been washed previously with alcohol, and dried. When separation has taken place, the alcoholic solution is drawn off, and the quantities of aldehyde, furfural, higher alcohols, esters, and acids contained in it are estimated, the usual methods being employed for this purpose. The author has ascertained by experiment that this treatment with oil does not introduce any error in the estimation of these compounds; he also finds that one extraction with olive oil is sufficient to remove the total quantity of "essences" present.

The process may be applied to the analysis of the liqueurs known as "aperitifs amers," the "essences" in which consist chiefly of orange oil; 1 grm. of this oil absorbs 3·42 grms. of iodine, and the amount of "essence" in a sample is calculated accordingly from the iodine absorption. W. P. S.

Detection of Benzoic Acid in Flesh and Fats. K. Fisher and O. Gruenert. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **20**, 580-583.)—The following process is described as being an improvement on the method recommended previously by the authors (ANALYST, 1909, **34**, 394). Fifty grms. of the sample (*e.g.*, butter) are shaken with 150 c.c. of a 1 per cent. sodium bicarbonate solution at a temperature sufficiently high to keep the fat in a molten condition. The aqueous solution, while still hot, is separated from the fatty layer and neutralised by the addition of sulphuric acid. Ten c.c. of Fehling's copper sulphate solution, and 10 c.c. of a 3·11 per cent. potassium hydroxide solution are added, and the mixture is filtered. If the mixture be only slightly acid or alkaline in reaction, a clear filtrate is always obtained. This filtrate is then acidified with sulphuric acid, and the benzoic acid is extracted with ether, the benzoic acid obtained being identified by various tests. For the latter purpose Mohler's test as modified by Halphen (ANALYST, 1908, **33**, 420) may be employed. The authors find that this test will not detect the presence of small quantities of benzoic acid in butter unless the benzoic acid has been separated in a pure condition, as above described. A test proposed by Jonescu (*J. Pharm. Chim.*, 1909, **29**, 523), in which the benzoic acid is converted into salicylic acid by the action of hydrogen peroxide, the salicylic acid formed being then detected by the addition of ferric chloride, is also trustworthy, and will detect as little as 0·006 per cent. of benzoic acid in butter. W. P. S.

Analysis of Meat-Juices. K. Micko. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **20**, 537-564.)—The present paper deals mainly with means for distinguishing between genuine meat-juices and those products which consist of mixtures of meat-juice, meat-extract, and egg-albumin. For the purpose of detecting the presence of egg-albumin in a sample of meat-juice, advantage is taken of the different temperatures at which meat-albumin and egg-albumin coagulate. The coagulating-point of meat-juice itself varies considerably, and may be as low as 40° C. It contains several different albumins, which coagulate at temperatures varying from 56° C. to 75° C., and other factors also have an influence on this point. As the proportion of salts present has a considerable effect on the coagulation of the albumins, the author recommends that the coagulating-point be determined after the addition of a definite quantity of salt such as ammonium sulphate. Treatment of the meat-juice with ammonium sulphate has the advantage that a clear filtrate is obtained after filtering the mixture, in which the coagulating-point of the albumin may be observed readily. Usually two points are observed when such a solution is heated, one at which a turbidity is developed, and another, at a somewhat higher temperature, when a flocculent precipitate forms. The author terms these the lower and higher coagulating-points of the mixture. When a solution of egg-albumin is mixed with an equal volume of a saturated solution of ammonium sulphate, and the mixture is treated with an equal volume of water (the whole is thus one quarter saturated with ammonium sulphate), the solution becomes turbid when heated to about 60° C., and a precipitate forms before the temperature reaches 70° C. In the case of meat-juices similarly treated, the higher coagulating-point is about 80° C. Another characteristic difference in the coagulating-points of the two albumins is observed when their solution is mixed with its own volume of a saturated ammonium sulphate solution, and a 4 per cent. solution of acetic acid is added. Egg-albumin remains quite clear for a period of fifteen minutes or longer, whilst meat-juice becomes turbid at once, and a precipitate forms after a time. If heat has been employed in the preparation of the meat-juice, the turbidity may not develop for a few minutes, owing to the smaller proportion of albumin present. The presence of meat-extract lowers the coagulating-point of meat-juice which has been treated with an equal volume of a saturated solution of ammonium sulphate, but the addition of an equal volume of water raises the coagulating-point once more to about 80° C. This fact allows mixtures consisting of meat-juice and meat-extract to be distinguished from preparations containing egg-albumin. Analyses are given of various commercial meat-juices. The results obtained show that real meat-juices contain but very small quantities of albumoses as compared with liquid preparations which consist essentially of diluted meat-extract. The species of animal from which any given meat-juice has been prepared may be more or less definitely determined by means of a serum test (ANALYST, 1904, 29, 11).

W. P. S.

Wendler's Method for the Estimation of Fat in Milk. F. E. Nottbohm and J. Angerhausen. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **20**, 495-498.)—The authors have investigated the "Neusal" method, described recently by Wendler (*Molkereizeit. Hildesheim*, 1910, **24**, 690), for the estimation of fat in milk. The milk is treated in a Gerber butyrometer tube with a proprietary saline solution and alcohol, and is then submitted to centrifugal action. The average difference between the results found by the new process and those of the Gottlieb method in the estimation of the fat in thirty-one samples of whole and skimmed milk was +0·045 per cent. The method works well with milks containing preservatives, the casein dissolving readily even in the presence of considerable quantities of formaldehyde, but there is considerable difficulty in the case of boiled milk. W. P. S.

Volumetric Estimation of Phenolphthaleïn. V. Zotier. (*Bull. Soc. chim.*, 1910 [iv.], **7**, 993-995.)—The following method of estimating phenolphthaleïn now so largely used in pharmacy is recommended: The weighed quantity (0·1 to 0·2 grm.) of the sample is triturated with a few fragments of sodium hydroxide (free from carbonate), the mixture dissolved in a few c.c. of water, and the solution filtered. The filtrate and washings are made up to 50 c.c., and 25 c.c. of the liquid treated with hydrochloric acid, which is added drop by drop until the phenolphthaleïn begins to precipitate. The precipitate is redissolved by the addition of the exact quantity of $\frac{N}{10}$ sodium hydroxide solution, and the solution titrated with $\frac{N}{10}$ sulphuric acid, with vigorous shaking after each addition of the acid. If n represent the number of c.c. of acid consumed, and v the volume of the solution at the end of the reaction, the amount of phenolphthaleïn in grams may be found by means of the expression—

$$2[(n \times 1{\cdot}59) + (v - n)0{\cdot}0092].$$

C. A. M.

Philippine Terpenes and Essential Oils. IV. R. F. Bacon. (*Philippine J. Science*, 1910, 5, 257-265.)—The following volatile oils are described:

Cinnamon Bark Oil (from bark of *Cinnamomum mindanaense*, Elmer).—This bark, which strongly resembles commercial (Ceylon) cinnamon in its characters, yields at least 0·4 per cent. of a yellow oil, of cinnamon-like odour, having the following characters: Sp. gr., 0·960 at $\frac{30°}{30°}$ C.; $[n]_{D\,30°C.}$ 1·5300; $[\alpha]_{D\,30°C.}$, +7·9°; cinnamic aldehyde content, 60 per cent.

Oil from Elemi Resin (from resin of *Canarium villosum*).—Eleven per cent. of volatile oil is obtained by distillation under reduced pressure. About 25 per cent. of the oil distils at 154° to 158° C., and the oil contains *d*-pinene in considerable quantity, dipentene (small amount), and *p*-cymene. The aqueous distillate contains formic acid. Different trees yield resins containing differing terpenes, pinene, cymene, and phellandrene being among the number.

Ginger Oil.—The Philippine ginger roots (from *Zingiber officinale*) yield 0·07 per cent. of a light yellow oil, having an odour of ginger and orange. Sp. gr. 0·885 $\frac{30°}{30°}$ C.; $[n]_{D\,30°C.}$, 1·4830, and $[\alpha]_{D\,30°C.}$ − 5·9°; saponification value, 14; soluble in two or more volumes of 90 per cent. alcohol.

Oil from Ocimum Sanctum.—The fresh leaves of this plant yield 0·6 per cent. of a green oil of anise-like odour, with sp. gr. 0·952 at $\frac{30°}{30°}$ C.; $[n]_{D\,30°C.}$, 1·5070; and saponification value, 2·8. The oil consists chiefly of methyl-homo-anisic acid (melting-point, 85° C.).

Zedoary Oil (from chopped roots of *Curcuma zedoaria*).—The roots obtained in September and December yielded the following oils:

	September.	December.
Yield, per cent.... ...	0·065	0·25
Colour	dark brown	light yellow
Sp. gr. at $\frac{30°}{4°}$ C.	0·933	0·993
$[n]_{D\,30°C.}$...	1·4920	1·5070
$[\alpha]_{D\,30°C.}$...	—	+1·10°
Saponification value ...	—	2
Solubility in alcohol ...	completely soluble in 2 volumes of 90 per cent.	completely soluble in 2 volumes of 80 per cent.

The oil contains a sesquiterpene alcohol of melting-point, 67° C.

Turmeric Oil (roots of *Curcuma longa*).—A yield of 0·22 per cent. of a brown oil was obtained, of the following character: Sp. gr., $\frac{30°}{30°}$ C., 0·930; $[n]_{D\,30°C.}$, 1·5030; $[\alpha]_{D\,30°C.}$, + 8·6°; ester number, 81; miscible with 75 per cent. or stronger alcohol in all proportions.

Champaca Oil (*Michelia champaca*).—Pure oils distilled from champaca flowers do not contain, according to the author, any benzoic acid, and oils obtained by Schimmel and Co. and containing benzoic acid were undoubtedly distilled from mixtures of flowers, probably with ylang-ylang in some cases. The crude oil (yield, 0·20 per cent.) on standing yields a large quantity of a solid substance, crystallising in white needles, melting-point 165°–166° C., and having the formula $C_{15}H_{20}O_{3}$. Iso-eugenol is also present in the oil. The portion of the oil soluble in ether, on treatment with 70 per cent. alcohol yields a neutral fraction of fine champaca odour, of sp. gr. (*a*) 0·9543, (*b*) 1·020, in two cases. Saponification value, 160 and 180.

Ylang-Ylang Oil.—The ester numbers of many oils distilled during the past season were from 130 to 150. (*Cf.* ANALYST, 1909, **34**, 32, 396.) A. R. T.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Oxydase and Reductase occurring in Cows' Milk. W. D. Kooper. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **20**, 564-575.)—The results of an investigation on the enzymes present in cows' milk.

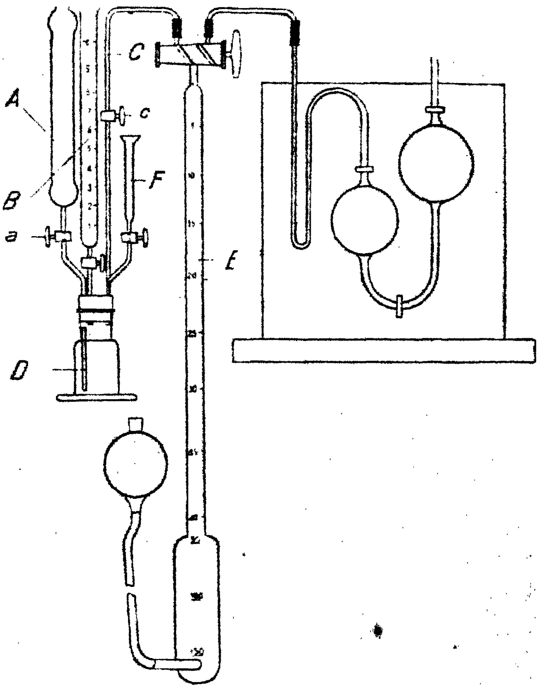
Testing the Porosity of Earthenware Filtering Bougies. F. Grenet. (*Compt. rend.*, 1910, **151**, 941-943.)—The earthenware filtering bougies which are employed for removing micro-organisms from liquids show great variations in their porosity according to the raw material and the mode of manufacture. This variability has an important bearing on their sterilising efficiency. The author has devised a method for comparing the structure of these bougies, depending on the capillarity phenomena produced when the porous bodies are immersed in water. The water penetrates forcibly into the interstices and, displacing the air, creates a pressure inside proportional to the fineness of the pores. A dry bougie in a vertical position is completely filled with mercury; a fine steel tube is then introduced through the orifice extending inside to the middle of the bougie and to a few centimetres outside. The tube is hermetically sealed in the orifice by means of shellac, and the free end of the steel tube is connected with a graduated glass tube of 1 mm. bore and a length of 1 to 2 metres. The apparatus being held in a vertical position, the bougie is completely immersed in water. The mercury is forced up the tube by the pressure of the air displaced in the capillary pores, and its height is observed. From this observation the diameter of the pores of maximum dimensions may be calculated. Commercial bougies occur which give columns of mercury ranging from 18 or 20 cm. up to 2 m. The force with which water enters a dry bougie is so great that micro-organisms may readily be carried through at first. In order to avoid this, it is advisable never to use a bougie in the dry state, but to steep it first in sterilised water before introducing the liquid to be filtered.

J. F. B.

Soda-Water. W. Collingridge. (*Report of the Medical Officer of Health for City of London to Sanitary Committee*, 1910, **141**, 140-145.)—Particulars of results of the bacteriological examination by Dr. Klein of thirty-six samples of soda-water are given. They show that, in the great majority of brands now offered to the public, *Bacillus coli* cannot be found in 10 c.c., and that the total number of microbes capable of growth at 37° C. is usually less than 10 per c.c., half of the samples showing less than 1 per c.c. On the other hand, three samples were swarming with bacteria capable of growth at 37° C., and showed *B. coli* in as little as 1 c.c. The Medical Officer concludes that there can be no serious difficulty in producing soda-water which is substantially sterile, and at all events free from excrementitious organisms, and suggests that the Local Government Board might reasonably draw up a standard of purity, and initiate a system of registration and inspection of mineral water factories, with a view to securing that all mineral waters shall be as wholesome as the majority are now shown to be. G. C. J.

ORGANIC ANALYSIS.

Quantitative Estimation of Aliphatic Amino Groups, and its Application in the Chemistry of the Proteins, Urine, and Enzymes. D. D. van Slyke. (*Ber.*, 1910, **43**, 3170-3181.)—Several attempts have been made to base a quantitative method upon the reaction of amino groups in aliphatic compounds with nitrous acid. The author claims that by the method now described all the previous objections have been overcome, and that an estimation may be made in a few minutes with an error not exceeding 0·05 mgrm. of nitrogen. The apparatus employed is illustrated in the accompanying figure. The glass tubes extending from the vessel, *D*, are all capillaries of 6-7 mm. external diameter, and, with the exception of that leading to *A*, of 1 mm. bore, the latter being 2 mm. The water in the gas burette, *E*, contains about 1 per cent. of sulphuric acid, in order to keep the glass clean. The reaction takes place in the bottle, *D*, of 35 to 37 c.c. capacity. The solution of the amino compound is first placed in the burette, *B*, containing preferably not more than 20 mgrms. of amino nitrogen; 5 c.c. of water are placed in *A*. Into the bottle, *D*, are introduced 28 c.c. of a solution of 3 parts of sodium nitrite in 10 parts of water, and then 7 c.c. of glacial acetic acid. Immediate development of nitric oxide takes place. The stopper carrying the tubes *A*, *B*, and *C* is then inserted and wired down, and the tap, *a*, is opened. Water enters and drives the air out from *D* through *C*. In order to expel the air dissolved in the nitrite solution, the tap, *c*, is closed, *a* is opened, and the bottle shaken; there is a powerful evolution of nitric oxide, and 10 to 15 c.c. of liquid are forced back into *A*. The tap, *c*, is again opened, and the gas containing the air is expelled through *C*. The operation is repeated. Ultimately



a volume of about 20 c.c. of gas is developed in *D* by shaking, the tap, *a*, is closed, and *C* is connected with the gas burette, *E*. The amino solution is then admitted to *D*, the reaction is completed by shaking, and the nitrogen, mixed with nitric oxide, is collected in *E*. The nitric oxide is absorbed in the Hempel pipette by means of an alkaline solution of permanganate, and the purified nitrogen is measured in *E*. The funnel, *F*, is only used when viscous solutions have to be tested, in which case it contains amyl alcohol, a few drops of which prevent frothing. The volume of air contained in the 10 c.c. of amino solution may be allowed for by deducting 0·16 c.c. from the observed volume of nitrogen. The volume of nitrogen evolved represents double that of the amino nitrogen reacting. Each of the natural amino acids evolves 1 molecule of nitrogen, with the exception of proline and hydroxyproline, which do not react, and lysine, which yields 2 molecules. In a mixture of the volatile esters of amino acids containing that of proline, the proline may be estimated by the difference between the total nitrogen and the amino nitrogen. In the analysis of the products of hydrolysis of proteins, the amino acids may be separated into two groups by means of phosphotungstic acid, and the amino nitrogen estimated in each. In the analysis of urine, after elimination of urea and ammonia, the method may be applied for the estimation of amino groups. Lastly, there is an obvious application of the method, in conjunction with estimations of total nitrogen, in studying the progressive hydrolysis of proteins by enzymes. Thus it is possible to calculate the degree (percentage) of hydrolysis, using the expression: $\frac{100\,(A - A^{\circ})}{A' - A^{\circ}}$ in which A = observed amino nitrogen, A° = amino nitrogen of the non-hydrolysed protein, and A′ = amino nitrogen after complete hydrolysis.

J. F. B.

Colour Reactions of Adrenaline and Allied Bases. A. J. Ewins. (*J. Physiol.*, 1910, **40**, 316-326; *Chem. Zentr.*, 1910, ii., 1569-1571.)—It is shown that the colour reactions produced by the action of iodine, mercuric chloride, and potassium di-iodate, on adrenaline are due to oxidation; a colour reaction described by the author, and depending on the red coloration produced when adrenaline is treated with potassium persulphate, is due to the same cause. This persulphate reaction is somewhat more sensitive than the other tests, and will detect the presence of 1 part of adrenaline in 5,000,000 parts of water. The amino base corresponding with adrenaline, dihydroxyphenylethylamine, the corresponding methyl-, ethyl-, and propylamine bases, and amino-acetopyrogallol, also give the reactions.

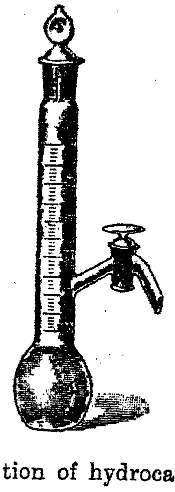
W. P. S.

Detection of Ethyl Alcohol in the Presence of Methyl Alcohol. G. Denigès. (*Bull. Soc. chim.*, 1910 [iv.], 7, 951-952.)—The method is based upon the fact that on treating methyl alcohol with bromine-water only traces of formaldehyde are formed, whereas ethyl alcohol under the same conditions yields an abundant quantity of acetaldehyde. In applying the test, 0·2 c.c. of the methyl alcohol is shaken with 5 c.c. of bromine-water (0·3 c.c. of bromine to 50 c.c. of water) in a long test-tube (about 20 cm. long and 25 mm. in diameter), which is then immersed in boiling water. If the yellow colour persists, the tube is left in the water for five or six minutes; otherwise it is removed as soon as the colour has practically disappeared. The tube is then cooled in water, and if its contents are still coloured, a dilute solution of sodium bisulphite (1 part of the commercial solution of sp. gr. 1·33 to 1·38 with 4 parts of water) is added drop by drop until decolorisation takes place, care being taken to avoid an excess. The liquid is then tested with 5 c.c. of Schiff's reagent (magenta just decolorised with sulphurous acid), and allowed to stand for at least five, but not more than eight, minutes. If ethyl alcohol was originally present, a red-violet colour is produced, the intensity of which is proportional to the quantity of alcohol. In the absence of that alcohol no appreciable coloration is obtained, since the product given by pure methyl alcohol under these conditions does not produce any coloration until after ten minutes' contact with the reagent. By the use of standard solutions of pure ethyl alcohol in methyl alcohol for comparison, the method may be made quantitative. For the detection of minute quantities of ethyl alcohol in dilute alcoholic solutions (0·05 to 3 per cent.), 5 c.c. of the liquid under examination are mixed with 0·2 c.c. of pure methyl alcohol, and 0·03 c.c. of bromine added by means of a capillary pipette. The tube is then shaken to dissolve the bromine, and placed in boiling water as described above. In this way it is possible to detect 2 mgrms. of ethyl alcohol. C. A. M.

Estimation of Ether and Benzene in Alcohol. H. Wolff. (*Chem. Zeit.*, 1910, **34**, 1193.)—For the estimation of ether in alcohol, the author describes the following modification of Holde and Winterfeld's method (ANALYST, 1908, **33**, 242): 100 c.c. of the spirit to be analysed are diluted to a sp. gr. of 0·96, and 20 c.c. are distilled over into a measuring cylinder of 100 c.c. capacity graduated in 0·5 c.c. The tube of the condenser should extend for some distance into the receiver (at least 2 to 3 cm.), and the latter should be well cooled. The distillate is treated with 80 c.c. of concentrated brine, and shaken to and fro about twenty times. After separation, the volume of the ethereal layer is read off, any bubbles which form at the line of separation being broken by turning and tapping the cylinder. The errors are generally less than 0·5 per cent. By this method spirits containing up to about 13 per cent. of ether may be analysed with accuracy, but if the percentage be higher, it is necessary to take only 50 or 25 c.c. of the spirit, dilute to 0·96 sp. gr., and distil off 20 c.c. A similar procedure is followed in the estimation of benzene in spirits, brine being employed instead of water, as prescribed by Holde and Winterfeld. The distillate is collected in a measuring cylinder of 30 c.c. capacity, graduated in $\frac{1}{10}$ c.c., or in a flask with graduated neck immersed in a bath of cold water. The separation of the benzene is sharper when brine is employed, and its volume may be read with an average error of only 0·1 c.c. J. F. B.

Estimation of the Bromine Value of Fats and Oils. Primary and Secondary Bromine Values. W. Vaubel. (*Zeitsch. angew. Chem.*, 1910, **23**, 2077-2080.)—In the course of some experiments on the estimation of the bromine values of oils, the author noticed that, when an oil is dissolved in carbon tetrachloride with the addition of potassium bromide and hydrochloric acid, and the solution is then titrated with potassium bromate solution, the quantity of bromine taken up by the oil is not equivalent to the iodine value of the oil. Further experiments showed that non-drying oils, such as olive oil, absorb little, if any, bromine when thus treated; whilst drying oils absorb the bromine readily up to a certain point, where the addition of a further quantity of bromate solution produces a yellow colour of free bromine in the solution. The quantity of bromine thus absorbed by the oil is termed the "primary bromine value." Only when an excess of bromine is added does the oil absorb a quantity equivalent to the iodine value, and this quantity is called the "secondary bromine value" of the oil. The following values were obtained in the case of three oils: Linseed oil, primary value, 75·2, 80·0; secondary value, 107·7. Wood oil, primary value, 57·0; secondary value, 94·6. Earthnut oil, primary value, 51·9; secondary value, 57·6. The author discusses the relation between these results and the compositions of the oils. For the determination of the total (secondary) bromine values of oils, the author recommends that 5 grms. of the oil be dissolved in 100 c.c. of carbon tetrachloride; potassium bromide solution and about 300 c.c. of water are added, then 10 c.c. of concentrated hydrochloric acid and a small crystal of potassium iodide. After the addition of an excess of standard potassium bromate solution, the mixture is shaken thoroughly for one minute, and the excess of bromine is then titrated with sodium sulphite solution. The small quantity of iodine added in the form of potassium iodide enables the end point of the titration to be observed clearly. The results obtained, when calculated into terms of iodine, agree closely with the iodine values of the various oils as estimated in the usual way.

W. P. S.



Estimation of Total Fatty Acids in Soap. A. Röhrig. (*Zeitsch. angew. Chem.*, 1910, **23**, 2161-2165.)—The use of the apparatus here shown simplifies and renders more accurate the estimation of total fatty acids in soap. From 1 to 2 grms. of the soap (or an aliquot portion of an aqueous solution of 5 to 10 grms.) are introduced into the apparatus, and the solution shaken with a slight excess of sulphuric acid. The liberated fatty acids are brought to the surface by gently heating and shaking the flask, and, when completely cold, are dissolved by the addition of 25 c.c. of a mixture of ether and petroleum spirit (boiling-point 35° C.) in equal parts, and the contents of the flask diluted with water to 100 c.c. An additional 25 c.c. of the mixture of ether and petroleum spirit is now added, and the flask vigorously shaken and allowed to stand. The volume of the ethereal layer is then noted, and one half of it drawn off into a tared basin and evaporated on a moderately hot water-bath until the residue of fatty acids becomes viscous, after which the basin is dried for ten minutes at 100° C. and weighed as soon as it is cold. In the case of cocoanut oil fatty acids, however, the temperature of drying must be kept at 57° to 62° C., concordant results being then obtained in the same time. The apparatus may also be used for the rapid estimation of hydrocarbons in commercial oleine. From 4 to 5 grms. of the sample are heated for ten to fifteen minutes on the water-bath with about 4 grms. of solid sodium hydroxide in about 30 grms. of 50 per cent. alcohol, and the liquid then cooled and made up to the mark with 50 per cent. alcohol. From 45 to 50 grms. of petroleum spirit (boiling-point 35° C.) are now added, in four equal portions, with vigorous shaking after each addition, and the apparatus is allowed to stand five or six hours, until the petroleum spirit layer becomes completely clear. One half is then drawn off and evaporated in a tared basin, and the residue dried for two to three hours at 60° to 70° C. In the case of American oleines the sum of the residue and of the free fatty acids estimated by titration is within $\frac{1}{4}$ to $\frac{1}{3}$ per cent. of 100. In certain French and German oleines, however, there is a difference due to the presence of neutral fat. In such cases 4 grms. of the oleine are titrated in the apparatus with $\frac{N}{1}$ alcoholic sodium hydroxide solution, the contents then made up to 100 c.c., and the estimation continued as described above. The residue from the petroleum spirit layer now consists of neutral fat and hydrocarbons, and the difference between this result and the first estimation gives the quantity of neutral fat. The apparatus may be obtained from F. Hugershoff, Leipzig. C. A. M.

Characteristics of Certain Oils. A. P. Lidoff. (*Chem. Rev. Fett- u. Harz-Ind.*, 1910, **17**, 262-263).—The seeds of *Ulmus campestris* yielded 26·09 per cent. of a non-drying oil, which was remarkable for containing volatile solid fatty acids and readily soluble non-volatile acids:

	Solidification-Point.	Sp. Gr.	Acid Value.	Saponification Value.	Hehner Value.	Reichert Value.	Iodine Value.	Acetyl Value.
	° C.							
Oil from *Ulmus campestris* seeds	– 3·5	0·9559 at 20° C.	5·57	277·3	75·45	3·75	32·2	—
Archangel shark-liver oil ...	—	0·9118 at 18° C.	12·10	158·3	98·80	1·20	127·7	17·2
Archangel seal oil	—	0·9294	9·80	191·9	95·20	0·00	119·4	12·0

C. A. M.

Characteristics of some Little-known Vegetable Oils and Fats. C. Grimme. (*Chem. Rev. Fett- u. Harz-Ind.*, 1910, **17**, 263-269; *cf.* ANALYST, 1910, **35**, 536).—The subjoined table gives the analytical values of oils and fats derived from the following plants: (1) *Allophyllus racemosus*, indigenous to Asia and Australia. The seed-kernels yielded 22·3 per cent. of a brown crystalline fat. (2) *Pentadesma butyracea* (West Africa), producing seeds that yielded 46·6 per cent. of a solid white fat. (3) *Garcinia balansæ* (India), from the seed-kernels of which 63·4 per cent. of a dark brown fluid oil was extracted. (4) *Calophyllum inophyllum* (Africa, Polynesia), the seed-kernels of which yielded 60·4 per cent. of a dark green oil, with slight drying properties. It contained 18·26 per cent. of resin. (5) *Bertholletia excelsa* (South America), producing nuts the kernels of which yielded 65·8 per cent. of oil (Brazil-nut oil). (6) *Poga oleosa*, the seed-kernels of which yielded 55·3 per cent. of a light yellow fluid oil. (7) *Telfairia pedata* (East Africa), from the seed kernels of which 60·75 per cent. of a fluid oil with pleasant taste and odour was separated. (8) *Telfairia occidentales* (West Africa). The seed-kernels yielded 48·2 per cent. of a thick viscous oil, with fairly good drying properties. (9) Species of *Citrullus* (South-West Africa), the seeds of which gave 11·8 per cent. of a light yellow fluid oil, with weak drying properties and pleasant taste. (10) Oil nuts from Singapore, probably derived from a species of *Lauraceæ*. The kernels yielded 38·5 per cent. of a grey-green crystalline fat.

No.	Oil or Fat.								Fatty Acids.					
	Sp. Gr.	Melting-Point.	Refractive Index.	Acid Value.	Saponification Value.	Iodine Value.	Hehner Value.	Unsaponifiable Matter.	Melting-Point.	Solidification-Point.	Refractive Index.	Neutralisation Value.	Iodine Value.	Mean Molecular Equivalent.
		° C.						Per Cent.	° C.	° C.				
1	0·8983 (75° C.)	63·0	1·4629 (60° C.)	136·70	242·2	39·4	95·76	2·31	62·0	60·5	1·4567 (60° C.)	226·5	39·9	248·0
2	0·9165 (30° C.)	42·0	1·4617 (25° C.)	26·40	197·0	42·3	96·10	0·92	57·0	54·5	1·4429 (60° C.)	198·7	42·8	279·7
3	0·9127 (15° C.)	8·6	1·4682 (40° C.)	19·90	176·3	86·2	93·18	4·23	32·5	30·3	1·4564 (60° C.)	161·8	89·1	341·4
*4	0·9415 (15° C.)	7·5	1·4772 (15° C.)	67·50	198·7	95·3	78·30	0·35	36·5†	32·5	1·4654 (45° C.)	192·3	94·8	289·0
5	0·9180 (15° C.)	—	1·4643 (25° C.)	79·40	202·0	98·3	96·82	0·85	29·5	27·0	1·4528 (45° C.)	196·7	99·2	286·6
6	0·9085 (15° C.)	−4·0	1·4700 (15° C.)	4·20	177·5	91·1	95·83	0·35	28·0	24·5	1·4499 (45° C.)	153·5	93·6	362·1
7	0·9185 (15° C.)	10·0	1·4686 (15° C.)	2·44	186·5	84·2	96·20	0·34	43·5	41·8	1·4492 (50° C.)	209·1	86·7	265·8
8	0·9135 (50° C.)	−1·0	1·4763 (20° C.)	61·54	262·2	43·4	96·20	0·38	42·0	39·5	1·4613 (50° C.)	221·0	45·9	251·5
9	0·9158 (15° C.)	−10·0	1·4751 (15° C.)	6·52	195·6	119·5	95·90	0·28	34·0	33·0	1·4568 (45° C.)	194·5	122·3	285·8
10	0·9021 (50° C.)	44·0	1·4549 (40° C.)	52·06	169·5	38·9	95·85	1·37	55·0	53·5	1·4424 (55° C.)	207·2	38·2	268·3

C. A. M.

* Containing 18·36 per cent. of resin.
† Without resin.

Separation of Solid from Liquid Fatty Acids. David. (*Comptes rend.*, 1910, **151**, 756-757.)—The method is based upon the author's observation that the ammonium salts of solid fatty acids are completely insoluble in a large excess of ammonia at 13° to 14° C., whereas the ammonium salts of liquid fatty acids are completely soluble therein. About 2 grms. of the mixed fatty acids are dissolved in 5 c.c. of 95 per cent. alcohol, the solution treated with 50 c.c. of ammonia solution at 22° C., and the mixture heated until bubbles of ammonia gas appear. The solution is now left for several hours at 14° C., and then filtered from the insoluble deposit of ammonium palmitate and stearate. The filter and its contents are washed with ammonia, until the filtrate ceases to give the slightest turbidity with baryta-water, after which a mixture of pure hydrochloric acid (sp. gr. 1·17) and water

1 : 1) is poured over the filter. The ammonium chloride solution which is formed passes through the filter, whilst the solid fatty acids are left. They are washed with water acidified with hydrochloric acid until free from ammonium salts, and then with water, detached from the filter, dried at 100° C., and weighed. In test experiments with a mixture of fatty acids containing 60 per cent. of solid acids, the amounts found were 59·7 and 59·8 per cent. Hydroxystearic acid, iso-oleic acid, lauric acid, and arachidic acid, also yield ammonium salts insoluble in a large excess of ammonia. The liquid fatty acids in the filtrate may be estimated by difference, or they may be liberated from their ammonium salts, washed, and dried at 120° C., with the usual precautions to prevent loss. C. A. M.

Separation of Oleic Acid from Stearic and Palmitic Acids. P. Falciola. (*Gazz. Chim. Ital.*, 1910, 40, 217-229.)—A method of separating oleic acid from stearic and palmitic acids is based upon the difference in solubility of their respective ammonium salts in alcohol, as is shown in the following results, obtained with specimens of the pure compounds:

SOLUBILITY IN GRMS. PER 100 C.C.

	Absolute Alcohol, ° C.					75 per Cent. Alcohol, °C.				50 per Cent. Alcohol, ° C.			
	50°.	40°.	30°.	20°.	10°.	40°.	30°.	20°.	10°.	40°.	30°.	20°.	10°.
Stearic acid	—	13·8	4·5	2·0	0·9	0·77	0·39	—	0·15	0·12	0·10	0·08 (23°C.)	—
Ammonium stearate	5·5	1·8	0·9	0·5	0·3	5·00	1·83	—	0·56	3·21	1·16	0·51	0·25 (12°C.)
Palmitic acid	—	31·9	—	9·2	2·8	3·59	1·19	0·43	0·24	0·31	0·12	0·09	0·05
Ammonium palmitate	11	4·5	—	1·4	0·7	14·84	11·02	4·33	1·78	6·69 (?)	—	5·33	—
Ammonium oleate	100	—	—	—	59	—	10·86	—	8·20	—	—	—	—

Ether dissolved 0·1 grm. of ammonium stearate, 0·29 grm. of ammonium palmitate, and 16·9 per cent. of ammonium oleate, per 100 c.c. at the ordinary temperature; whilst in acetone there dissolved, per 100 c.c., 0·08 grm. of ammonium stearate at 13° C., 0·20 grm. of ammonium palmitate at 13° C., and 4·7 grms. at 15° C. For a quantitative separation of these fatty acids the mixture is dissolved in warm ether, and the solution treated with ammonia gas, and cooled to the ordinary temperature. The solvent is then evaporated, and the residue thoroughly mixed with about four times its volume of ammoniacal alcohol at about 0° C., and filtered with the aid of a pump. The residue is washed with the smallest possible quantity of chilled absolute alcohol, the filtrate and washings treated with dilute hydrochloric acid, the solvent evaporated, and the liberated liquid fatty acids washed, dried, and weighed. The solid fatty acids upon the filter are also decomposed with hydrochloric acid, and estimated in the usual manner. The following results illustrate the degree of accuracy thus obtainable:

Taken.	Grms.	Found.	Grms.
Oleic acid ...	1·92	Oleic acid ...	2·08
Palmitic and stearic acids ...	1·28	Solid fatty acids ...	1·18
	3·20		3·26
Oleic acid ...	4·95	Oleic acid ...	4·89
Palmitic acid ...	2·55	Solid fatty acids ...	4·97
Stearic acid ...	2·48		
	9·98		9·86

C. A. M.

Iodine Values of Linoxyn. Meister. (*Chem. Rev. Fett- u. Harz-Ind.*, 1910, **17**, 260-262.)—Glacial acetic acid (98 per cent.) dissolves linoxyn, and is therefore the best solvent for estimating the iodine value of that substance. Old varnish films are not completely soluble in the cold acid, and gentle heat is required to dissolve them. The older the linoxyn, the greater the tendency to the formation of a slight residue insoluble in glacial acetic acid. Hence it is not possible to follow the drying process of linseed oil completely to the end, although the iodine values may be estimated to a stage at which no further material increase in weight by oxidation takes place. The hot glacial acetic acid does not have any appreciable effect upon the results, as is shown by the fact that linseed oil fatty acids gave the same iodine value whether dissolved in chloroform or in hot glacial acetic acid. The following results were thus obtained with two samples of boiled linseed oil, the iodine values (Hübl) in the sixth column being only approximate, owing to the separation of traces of insoluble matter towards the end of the titration:

	Original.	After 2 Hours' Drying.	After 4 Hours.	After 6 Hours.	After 8 Hours.	After 10 Hours.	After 12 Hours.
Boiled Oil I.:							
Iodine value ...	153·8	131·6	105·0	73·9	47·3	26·9 (?)	—
Increase in weight per cent.	—	5·4	10·2	16·8	17·9	18·5	18·6
Boiled Oil II.:							
Iodine value ...	156·8	136·1	104·0	74·8	48·8	29·1 (?)	—
Increase in weight per cent.	—	5·1	10·0	16·2	17·4	18·0	18·1

It was not possible to examine tung oil varnish in this way, as after four hours' drying the moist film was insoluble in glacial acetic acid and all other solvents.

C. A. M.

Acids Suitable for Use as Standards in Acidimetry. J. H. Kastle. (*Amer. Chem. J.*, 1910, **44**, 487-493.)—The author recommends three organic acids of the aromatic series which are eminently suitable for use as standards in acidimetry. These bodies are *p*-nitrotoluene sulphonic acid, *p*-amino-*o*-sulphobenzoic acid, and monopotassium *o*-nitro-*p*-sulphobenzoate. They are all well-defined substances, easily prepared in a perfectly pure condition. The two last are anhydrous, and can therefore be thoroughly dried by any convenient means without the danger of alteration in composition. The first, *p*-nitrotoluene sulphonic acid, crystallises with 2 molecules of water, but the crystals are very stable under ordinary atmospheric conditions. They all yield sharp end reactions on titration with phenolphthaleïn. For all practical purposes, the quantity of sodium hydroxide required for neutralisation coincides closely with the theoretical quantity, whilst with *p*-amino-*o*-sulphobenzoic acid the agreement is such as to meet the most exact requirements of volumetric analysis.

J. F. B.

The Alcohol-Soluble Constituents of Litmus. P. Scheitz. (*Zeitsch. anal. Chem.*, 1910, **49**, 736-739.)—The alcohol-soluble constituents of litmus, which are rejected in the course of the usual process of purification, include a small quantity of a colouring matter which is a more sensitive indicator than azolitmin. Dilute hydrochloric acid is added to powdered litmus until no more carbon dioxide is evolved and the liquid is distinctly red. The mixture is heated on the water-bath until a dark brown precipitate separates. This is filtered off, well washed with hot water, dried, and reduced to a fine state of division. A kilogramme of good litmus will yield from 120 to 180 grms. of this powder, which contains most of the valuable constituents of the original litmus, both the azolitmin and the alcohol-soluble colour. The finely-divided substance is boiled for half an hour with 4 to 5 litres of water, and then allowed to stand an hour, when the supernatant liquor is syphoned off, carrying with it a quantity of a very finely divided red substance, which will remain in suspension for weeks. Washing by decantation is repeated until, after standing an hour, the supernatant liquor is seen to be free from particles of the finely divided red substance. The liquors containing the red substance in suspension are evaporated to dryness, and yield 20 to 25 grms. of substance, about half of which is soluble in warm dilute alcohol. The residue from the elutriation experiment contains most of the azolitmin, contaminated, however, by a small quantity of the alcohol, soluble substance. Both the crude azolitmin and the product separated from it by elutriation are boiled for half an hour with 60-65 per cent. alcohol and the solution filtered hot. The extraction with dilute alcohol is repeated three or four times. The alcoholic extracts deposit a gelatinous precipitate on cooling. This is washed with aqueous alcohol, dried, and extracted with acetone, to remove erythrolein and erythrolitmin. Finally, the product is freed from inorganic matter by dissolving again in hot 60-65 per cent. alcohol, filtering hot, and allowing to cool, when it separates as a gelatinous, ash-free substance, of which 15 grms. may be obtained from 1 kilogramme of litmus. When ammonia gas is led over this substance it takes up some 8 or 9 per cent. of its weight of ammonia very rapidly. The ammonium salt, freed from excess of ammonia in a vacuum desiccator, is a sensitive indicator. One c.c. of a 0·1 per

cent. solution imparts a colour of convenient intensity to 100 c.c. of water, and the tint of this coloured water is changed sharply from red to blue by 0·4 c.c. of $\frac{N}{100}$ caustic potash, and back again to red by 0·3 of $\frac{N}{100}$ hydrochloric acid. Azolitmin forms a similar ammonium salt; and it is said that the azolitmin of commerce, which yields 8·5 per cent. of ammonia on warming with caustic alkali, is such an ammonium salt.
G. C. J.

Sensitive Indicator. J. F. Sacher. (*Chem. Zeit.*, 1910, **34**, 1192-1193.)—An extremely sensitive indicator is obtained by extracting the colouring matter from the fresh, undried skins of red radishes by means of 96 per cent. alcohol in the cold. The filtered extract possesses a dull-red colour, which, however, is more intense if traces of plant acids be present. In the latter case a little alkali is added until the coloration is less pronounced. This extract, when used as an indicator, gives a red coloration with acids, more intense than that afforded by methyl orange. With alkalis it gives a green coloration, which is blue at higher concentrations. The indicator is more sensitive to alkalis than methyl orange or phenolphthaleïn; 2 c.c. of the alcoholic extract showed a distinct green coloration with 15 c.c. of water containing 15 parts of calcium carbonate per 100,000. In titrations it gives a sharp change; it is preferable to titrate to the red point. Like litmus and phenolphthaleïn, it is sensitive to carbonic acid.
J. F. B.

Method for Purifying and Drying Organic Liquids by Wiping. C. L. Jackson and A. H. Fiske. (*Amer. Chem. J.*, 1910, **44**, 438-441.) — In the course of a research, the authors obtained a very small quantity of an oily liquid, far too small to dry and distil in the ordinary way. Since it was also volatile, it could not be dried in a desiccator. It was found possible to get it sufficiently dry to serve for elementary analysis, by merely wiping it with filter-paper strips which were previously moistened slightly to prevent their taking up the oil. The method was then tried on substances of known composition, such as wet chloroform containing 0·2 per cent. of iodine. It was found possible to remove the whole of the iodine from this solution by means of filter-paper strips moistened with potassium iodide solution. When 0·5 grm. chloroform was taken for the test, not much more than 0·2 grm. remained by the time the iodine and the last of the wash-water had been removed; but this small quantity was sufficient for analysis, which showed that the chloroform contained less than 0·5 per cent. of water and no other impurity.
G. C. J.

Valuation of Logwood. G. Grasser. (*Collegium*, 1910, 461-463.)—An approximate valuation of logwood may be made by extracting a weighed quantity (4 to 6 grms.) of the ground wood (containing 10·5 per cent. of moisture) with 200 c.c. of water for one and a half hours, diluting the solution to 250 c.c., and treating an aliquot portion (50 c.c.) with 20 c.c. of a 20 per cent. solution of lead acetate. The volume of the precipitate (separated with the aid of centrifugal force) is then noted, and its amount compared with that obtained under parallel conditions with a standard sample of logwood.
C. A. M.

Critical Investigations on the Analytical Methods for Raw Rubber. O. Korneck. (*Gummi-Zeit.*, 1910, **25**, 4-9, 42-46, 77-88; through *J. Soc. Chem. Ind.*, 1910, **29**, 1321.)—Working under the direction of Harries on eleven different sorts of rubber, the author has compared the various methods which have been proposed for the analysis of raw rubber. He concludes that—(1) Spence's method furnishes very similar results to the modified nitrosite method (see below). (2) The tetrabromide method (Budde, *Gummi-Zeit.*, 1909, **24**, 4) in its present form does not yet appear suited for the quantitative determination of rubber. (3) All other methods examined cannot be compared with the above. Spence's method consists essentially in the estimation of resin by complete extraction with acetone, determination of insoluble matter by solution in benzene without limitation of time, and estimation of rubber by evaporation of the filtered solution of the resin-free rubber. The methods mentioned in (3) include those of Schneider (*J. Soc. Chem. Ind.*, 1903, **22**, 1885), Fendler (alcohol precipitation, *ibid.*, 1904, **23**, 1117, modified by the use of carbon tetrachloride as solvent, as recommended later by Fendler and Kuhn), and Fendler-Harries nitrosite method (*Ber. deut. Pharm. Ges.*, 1904 [5]). The author especially emphasises the importance of treatment till the weight remains constant, instead of for a specified time, in both vacuum drying and acetone extraction. It is shown that the resins must be quantitatively removed in the Fendler-Harries method. Fendler prescribes six hours' extraction, but the author urges that this is not sufficient for all rubbers. The new form of Harries' nitrosite method which obviates this difficulty is as follows: 1 grm. of finely divided technically pure rubber (best dried and acetone extracted) is dissolved as far as possible in 75 c.c. of benzene in a beaker. Nitrous fumes are passed in till a dark green colour is obtained, stirring meanwhile to break up the precipitate. After settling a moment the clear green solution is decanted through a Gooch crucible. The precipitate is washed with benzene several times, 75 c.c. of benzene are added, and nitrous fumes are again passed. The beaker and leading tube, covered with a clock glass, are allowed to stand for twenty-four hours. The precipitate is kneaded together, pressed on the walls of the beaker, the clear green solution decanted through the Gooch crucible, and the precipitate washed with benzene till the filtrate is colourless. The nitrosite is then transferred to the crucible and washed with absolute ether till a homogeneous, fine bright yellow powder is left. The beaker, tube, nitrosite, and crucible, are dried for half an hour *in vacuo* and then at 80° C. in an ordinary oven to constant weight. 50 c.c. of acetone are then placed in the beaker, which is warmed on the water-bath till nothing more dissolves. The liquid is filtered through the Gooch crucible, and the precipitate washed with acetone. The beaker, tube, and crucible, are again dried to constant weight; the difference gives the weight of nitrosite. Pure rubber = nitrosite × $\frac{136}{259}$. The nitrous fumes are prepared from nitric acid (sp. gr. 1·3) and arsenic trioxide in pieces the size of hazel-nuts. The flask, provided with a dropping funnel, is warmed in an air-bath at first, and afterwards cautiously with a small flame. The gas is passed through a washing-flask to remove nitric acid, and is then dried over phosphorus pentoxide. The values obtained for pure rubber by this method, when added to those for insoluble matter (Spence's method) and resins (in this case determined by a new method using ozone), approximate very closely to

100 per cent., whereas the values for pure rubber by Budde's method are from 1·5 to 10 per cent. lower.

Starch-Iodine Reaction. W. Harrison. (*Proc. Chem. Soc.*, 1910, **26**, 2523.)—The author finds that in the formation of the blue iodide of starch the presence of iodides is not necessary. The blue colour is formed with pure iodine, but the depth is increased by the presence of any salt or weak acid. The gradual addition of alcohol changes the blue to violet, red, orange, and yellow, and the reverse change takes place on dilution with water. The same colour change is produced by carefully heating the blue solution, and the reverse phenomena are observed on cooling. This colour change is analogous to the colour change of colloidal gold solution, investigated by Zsigmondy, which changes from red to blue on adding salts.

The author succeeded in preparing a blue colloidal solution of iodine in water containing glycerol and no starch, but the colour only remained for a fraction of a second, the iodine being precipitated.

It was shown that all the iodine can be removed from starch iodide by washing with pure water, and the conclusion is drawn that the blue solution of iodine and starch is a colloidal solution of iodine, the starch acting as a protective colloid. It was further shown that the reactions of dextrins and starch celluloses can be obtained with starch under certain conditions, from which the conclusion is drawn that dextrins and starch celluloses are all different colloidal states of starch, and not chemically different compounds.

New Method of Estimating Sucrose in the Presence of Other Sugars. A. Jolles. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **20**, 631-638.)—The process described depends on the fact that the optical activity of practically all sugars, other than sucrose, is destroyed when the sugars are heated in dilute solution with sodium hydroxide. The sugars thus affected are arabinose, rhamnose, dextrose, lævulose, galactose, mannose, invert sugar, maltose, and lactose. The conditions governing the reaction are simply that the solution shall not contain more than 2 per cent. of these sugars, and that the alkalinity of the solution shall be approximately one-tenth normal. The destruction of the optical activity is brought about by heating such an alkaline solution to boiling for forty-five minutes either under a reflux condenser or in a pressure flask; the same change is affected by maintaining the mixture at a temperature of 37° C. for twenty-four hours, and this procedure has the advantage that the solution does not become dark in colour. The concentration of the sucrose is without effect on the accuracy of the process. As applied to the estimation of sucrose in wine, the details of the process are as follows: 100 c.c. of the wine are neutralised, evaporated to a volume of 50 c.c., treated with 5 c.c. of lead acetate solution, diluted with water to a volume of 100 c.c., and filtered. Twenty c.c. of the filtrate are then treated with sodium phosphate to remove the excess of lead, filtered, and sufficient sodium hydroxide (16 per cent.) solution is added to render the alkalinity of the mixture approximately one-tenth normal. The mixture is then heated as above described, and, after cooling, the polarisation is determined. If the mixture has been boiled, it may be treated with a further quantity of 5 c.c. of lead acetate solution and filtered before being examined in the polariscope, a correction being made for the dilution thus caused. The polarisation of the solution is now due solely to sucrose, and the quantity of the latter present in the wine is found by a simple calculation. In the case of condensed milk, 10 grms. of the sample are dissolved in water, lead acetate solution is added, and, after the excess of lead has been removed by means of sodium phosphate, the solution (filtrate) is diluted to a volume of 200 c.c. A portion of this solution is then heated as described after the addition of the requisite quantity of sodium hydroxide, and the polarisation of the solution is determined subsequently. The results obtained agree closely with those yielded by the ordinary polarimetric method for estimating sucrose.

W. P. S.

Examination of Wool-Grease Oleines. J. Marcusson. (*Mitt. Königl. Materialprüfungsamt*, 1910, **28**, 469-471.)—Wool-grease oleine contains little or no cholesterol, for, on distilling the unsaponifiable matter under reduced pressure, the whole of the fractions are dextro-rotatory, whilst their rotation steadily rises. Moreover, the original dextro-rotation is not changed by long-continued extraction with acetic anhydride, as would be the case in the presence of any notable proportion of cholesterol. Absorption of oxygen from the air by unsaturated bodies is quite sufficient to account for the presence of oxygen in the unsaponifiable matter of wool-grease oleine. Samples of English wool-grease oleine examined by the author had approximately the same characteristics as samples of French or German origin, although in one case the iodine value of the former was somewhat lower. Two samples were found to contain 48 and 54 per cent. of unsaponifiable matter, the iodine values of which were 65·4 and 51·3, and the optical rotation $[\alpha]_D = +24{\cdot}8^\circ$ and 24·1° respectively. As a rule, pure wool-grease oleine is soluble in its own volume of 96 per cent. alcohol at 20° C., whereas in the presence of 20 per cent. (or in some instances less) of mineral oil the liquid remains turbid. The optical rotation of pure wool-grease oleine of different origin was found to range from $[\alpha]_D = +10^\circ$ to 19°. If a sample has a rotation exceeding $[\alpha]_D = +10^\circ$, and gives a clear solution in the solubility test, it cannot contain any notable quantity of mineral oil. If, however, the presence of the latter be indicated by either of these tests, a further examination of the unsaponifiable matter must be made. C. A. M.

Detection of Mineral Oil in Wool-Grease Oleine. G. Winterfeld and W. Mecklenburg. (*Mitt. Königl. Materialprüfungsamt*, 1910, **28**, 471-474.)—By increasing the proportion of solvent in Marcusson's alcohol test (preceding abstract) to one and a half times the volume of the oleine it is possible to detect 10 per cent. and in some cases 5 per cent. of mineral oil. A still more sensitive test is to treat the sample with an equal volume of a mixture of 90 per cent. methyl alcohol and 10 per cent. of ordinary alcohol, a turbidity and ultimate separation of an oily layer taking place at 20° C. in the presence of 10 per cent. and in most cases of 5 per cent. of mineral oil, whilst pure wool-grease oleine usually gives a clear solution. In the case of certain samples of English wool-grease oleine, however, a mixture of oil and solid substances separated in this test, although in other respects the samples appeared pure. This abnormal behaviour is to be attributed to the very high proportion of solid unsaponifiable matter: and in this connection it is mentioned that very little wool-grease oleine of English origin is used in Germany, owing to the difficulty of removing it subsequently from the material. It is also stated that it is difficult to obtain English samples that have not been adulterated. C. A. M.

Detection of Rosin Oils in French Wool-Grease Oleines. G. Winterfeld. (*Mitt. Königl. Materialprüfungsamt,* 1910, **28**, 474-476.)—The methyl-ethyl alcohol solubility test (preceding abstract) enables an addition of 20 per cent. and in some cases of 10 or 5 per cent. of refined rosin oil to be detected in wool-grease oleine, a turbidity or separation of oil being obtained after the mixture has stood for an hour at 20° C. This test, however, usually fails to detect the presence of even 20 per cent. of crude rosin oil (rich in colophony). An addition of 10 per cent. or in some instances of 5 per cent. of either crude or refined rosin oil produces a material increase in the refractive index, which in the case of pure French wool-grease oleine is usually less than 1·480 at 20° C. C. A. M.

INORGANIC ANALYSIS.

Elimination of Ammonium Salts prior to Estimation of Alkali Metals. J. Jámbor. (*Zeitsch. anal. Chem.,* 1910, **49**, 733-734.)—The following method is recommended for the removal of large quantities of ammonium salts. The solution is evaporated on the water-bath until a crust of crystals appears. It is then allowed to cool, half its bulk of concentrated sulphuric acid is added, and the solution evaporated to dryness over a small flame. In this way the ammonium salts can be almost completely eliminated without attention or fear of creeping or spirting. G. C. J.

Detection and Estimation of Arsenic Acid in Presence of Arsenious Acid by Means of Magnesia Mixture. J. C. Brünnich and F. Smith. (*Zeitsch. anorg. Chem.,* 1910, **68**, 292-296.)—This method having been discredited by the work of Lutz and Swinne (Analyst, 1910, **35**, 76), the authors have re-investigated the conditions which determine the precipitation of arsenic acid whilst leaving arsenious acid in solution. They conclude that arsenic acid can be estimated in presence of arsenious acid by means of magnesia mixture, provided (*a*) the solution does not contain much more than 0·5 grm. As_2O_3 in each 100 c.c., (*b*) alkali salts are not present in greater quantity than corresponds to 1 per cent. NaOH, and (*c*) the magnesia mixture used contains in each 100 c.c. not more than 1·4 grms. NH_3 nor less than 10 grms. NH_4Cl, together with the usual 5·5 grms. of crystallised magnesium chloride. Experiments are described which show that if the addition of ammonia, recommended by Fresenius, Treadwell, and others, be avoided, and a magnesia mixture of the above composition (*i.e.*, with less ammonia than the usual reagent) be used, the method as a qualitative test is rendered more sensitive. The quantitative results show *minus* errors of the order of 1 per cent., and these errors increase greatly when alkali salts are present in quantity. G. C. J.

Estimation of Carbon and Sulphur in High-Grade Alloys of Tungsten, Molybdenum, and Vanadium, with Iron. E. Müller and B. Diethelm. (*Zeitsch. angew. Chem.*, 1910, **23**, 2114-2124.)—Experiments are described which show that, in alloys containing large proportions of tungsten, molybdenum, or vanadium, carbon and sulphur can be estimated accurately only by the direct dry combustion method. Wet combustion methods depending on the use of chromic and sulphuric acids, which give excellent results with ordinary steels, may, in the case of such alloys as have been named, lead to the carbon being underestimated to the extent of 20 to 80 per cent. The error in a sulphur determination made by a wet process is very much less, but the dry method is preferred both on the ground of accuracy and because the determination can then be combined with that of carbon. G. C. J.

Estimation of Carborundum in Coke Crucible Scrap. H. Wdowiszewski. (*Chem. Zeit.*, 1910, **34**, 1209-1210.)—At the temperature of the Siemens crucible furnace, notable amounts of carborundum may be formed in crucible bodies into the composition of which coke, as distinguished from graphite, has entered. Since the proportion of carborundum in new crucibles has a marked influence on their behaviour and life, it is important to be able to estimate the proportion present in old crucibles which it is proposed to incorporate with new materials. No direct method is found to be so satisfactory as the following indirect one: Uncombined carbon is estimated by ignition to constant weight. The residue is fused with alkali carbonate, and silica, alumina and iron oxide, lime and magnesia, estimated with the utmost accuracy attainable, since the sum of all the errors will fall ultimately on the carborundum, the quantity of which it is wished to estimate. Potash and soda may be estimated, but in any one works their amount will be found to approximate a constant, in the author's works 0·5 per cent. The results of many analyses of new crucible bodies made by the author add up to 99·5 per cent. in nearly every case, so that he regards 0·5 per cent. as a proper allowance for error in analysis. In the case of an analysis of material containing carborundum, the results of analysis add up to more than 100 per cent., since, for every 40 parts of SiC, 60 parts of SiO_2 are weighed. The results of analysis, *plus* 0·5 per cent. (unavoidable analytical error), are therefore summed; and if the sum be represented by $100 + x$, then the percentage of carborundum in the original material is given by $2x$. G. C. J.

General Method for the Preparation of Anhydrous Chlorides, and its Application in Chemical Analysis. F. Bourion. (*Annal. Chim. Phys.*, 1910, **20**, 547-573; *Chem. Zentr.*, 1910, ii., 1360-1361.)—The method consists in heating metals or metallic oxides in a current of sulphur chloride or a mixture of sulphur chloride and chlorine. For the preparation of thorium, chromium, and uranium chlorides and tungsten oxychloride, the use of sulphur chloride (S_2Cl_2) is recommended; whilst the mixture of sulphur chloride and chlorine is more suitable for the preparation of silicon, zirconium, and beryllium chlorides. The chlorides of many of the metals of the rare-earth groups may be prepared by this method.

Separation of Tungsten from Silica.—The process depends on the fact that tungstic acid is completely volatilised when heated to a temperature of 150° to 280° C. in a current of sulphur chloride and chlorine, whilst precipitated silica is not volatilised or attacked at temperatures below 525° C.

Separation of Cerium, Thorium, and Beryllium.—The mixed oxides are placed in a quartz boat, which is then heated in a silica tube to a temperature of 700° to 780° C., while a current of sulphur chloride and chlorine is passed through the tube; the thorium and beryllium chlorides are volatilised, whilst the cerium chloride remains in the boat. The condensed volatile chlorides are allowed to drop into hot sodium hydroxide solution, which is stirred meanwhile; thorium hydroxide is thus precipitated, whilst the beryllium remains in solution. After cooling, the mixture is diluted with water, filtered, and the thorium hydroxide is washed with dilute sodium hydroxide solution. The thorium hydroxide is then dissolved in acid, and both it and the beryllium are precipitated with ammonia from their solutions.

W. P. S.

Citrate-Solubility of Phosphoric Oxide in Basic Slag. E. V. Flack. (*Trans. Cape Chem. Soc.*, Aug. 26, 1910.) — The author shows that, as is well recognised, there is a large difference between the proportion of phosphoric acid dissolved from basic slag by Petermann's neutral ammonium citrate solution and the proportion dissolved by 2 per cent. citric acid solution, the latter being the solution chiefly used in Europe for determining the proportion of presumably readily available phosphoric acid in this material; and he recommends that the latter process should be used in South African laboratories, in order that the results obtained may be comparable with those of English and Continental chemists. The author points out that, in analysing the citric acid solution of the slag, if the phosphoric acid be determined by "direct" precipitation with magnesia mixture in presence of ammonium citrate, reliable results cannot be obtained unless the silica is first removed by evaporation of the solution to dryness with strong hydrochloric acid. Unless this precaution is observed, the percentage of soluble phosphoric acid indicated is, in his experience, on the average about 0·5 per cent. too high.

Electrolytic Separations. I. H. Buckminster and E. F. Smith. (*J. Amer. Chem. Soc.*, 1910, **32**, 1471-1477.)—Information is given concerning some twenty electrolytic separations, as to which the literature is conflicting, but which are shown to be quite practicable under assigned conditions. As the result of nearly a hundred experiments, the following directions are given for the electrolytic separation of silver and bismuth: To a solution containing not more than 0·2 grm. silver or 0·2 grm. bismuth as nitrate, 3 c.c. of nitric acid is added, and the mixture diluted to about 75 c.c. A dish anode is employed, and placed 3 mm. from the dish cathode. The liquid is maintained at a temperature of 60° C., and the anode rotated about 250 times a minute. At a pressure of 3 volts and current $\text{N.D.}_{100} = 0{\cdot}15$ to 0·20 ampère, 0·1 grm. of silver may be deposited quantitatively and free from bismuth in twenty-five minutes. The addition of chrome alum or iron alum to the electrolyte is not only unnecessary, but actually disadvantageous.

It is found that zinc may be separated from uranium by the use of a mercury cathode and rotating anode. The latter is conveniently given 200 turns per minute. With a current of 3·5 ampères at 5 volts, 0·2 grm. of zinc may be separated from an equal weight of uranium in from fifteen to sixty minutes. G. C. J.

Electrolytic Determination of Chlorine in Hydrochloric Acid with the Use of a Silver Anode and Mercury Cathode. J. S. Goldbaum and E. F. Smith. (*J. Amer. Chem. Soc.*, 1910, **32**, 1468-1471.)—Gooch and Read have stated (*Amer. J. Science*, 1909, 544) that when attempts are made to determine chlorine in hydrochloric acid with the use of a silver anode, results are obtained which are very irregular and always low. Gooch and Read used a platinum cathode. It is now shown that when a mercury cathode is used, results can be obtained of a high order of accuracy. The silver anode is prepared by plating platinum gauze from a potassium silver cyanide electrolyte. If this be then dipped in dilute hydrochloric acid, washed and ignited to incipient redness, any admixed cyanide is destroyed. The anode must not be strongly ignited, lest the silver be fused or its porous nature destroyed. Porosity favours retention of chlorine, whereas a smooth silver surface introduces a risk that molecular chlorine may be liberated, with consequent production of hypochlorites and chlorates. The deposit of silver chloride is liable to be contaminated by silver oxide due to a secondary action; but this secondary action involves no loss of chlorine, and the silver oxide is easily decomposed at temperatures above 250° C. For this purpose the authors use an electric oven, which can be maintained at 300° C., ten to fifteen minutes at this temperature being sufficient. It is convenient to rotate the anode some 300 times a minute, and high current densities must be avoided, lest the silver chloride become too loosely adherent. At the pressures usually employed (2·5 to 5 volts) the anode should be placed at least 15 mm. from the level of the mercury. The method is equally applicable to hydrobromic acid. G. C. J.

Analysis of Enamel. R. D. Landrum. (*Chem. Engineer*, 1910, **12**, 142-145.)—A detailed scheme of analysis for enamel is given, including the determination of silica, iron, alumina, lime, magnesia, alkalis, boron, fluorine, manganese, cobalt, antimony, tin, phosphorus, and lead. Little originality is claimed for the methods themselves, but only for their adaptation to the very difficult problem of enamel analysis.

The following method is described for stripping the several films of which an enamel is usually composed, and which must be analysed separately if useful results are to be obtained. The article is scratched lightly with emery-cloth or a file, and then coated with gum acacia or glue. The article is next heated in an air-bath and the hardened glue chipped off, carrying with it some of the outer coat of enamel. The glue is dissolved in water and the enamel recovered for analysis, and the process is repeated several times, to strip first the remainder of the outer coat and subsequently enough of the ground coat for analysis. G. C. J.

Estimation of Gold by Means of Hydrogen Peroxide. L. Rössler. (*Zeitsch. anal. Chem.*, 1910, **49**, 739-740.)—Vanino and Seemann (*Ber.*, 1899, **32**, 1968) showed that gold could be precipitated quantitatively in the cold and in a few minutes by means of hydrogen peroxide in presence of caustic alkali. It is now shown that precipitation is complete in acid solution in something less than twenty-four hours, and that, in presence of potassium carbonate in place of caustic alkali, it is not only complete, but rapid. G. C. J.

Delicate Test for Hydrogen. C. Zenghelis. (*Zeitsch. anal. Chem.*, 1910, **49**, 729-732.)—The gas, previously freed from other gases which might give the same reaction, is passed through a solution of sodium molybdate contained in a test-tube, in which is also placed a piece of recently-ignited platinum (or, better, palladium) gauze or foil. A single bubble of pure hydrogen produces an intense blue colour if the solution be warm; whilst, if it be cold, a greenish-blue colour results, and the same greenish-blue colour results on leading in a stream of gas containing traces only of hydrogen. The molybdate solution is made by dissolving 1 grm. of molybdenum trioxide in dilute caustic soda, rendering just acid with hydrochloric acid, and diluting to 200 c.c. If intended for use with palladium, the smallest possible excess of acid must be used. The gas to be tested must not only be freed from reducing gases, such as hydrogen sulphide and P_2H_4, but also from carbonic oxide. Neither methane, ethylene, acetylene, nor their homologues interfere. Methods and apparatus are described for concentrating on palladium sponge the hydrogen from large quantities of gas containing only minute traces of hydrogen, and subsequently demonstrating the presence of this hydrogen by means of the molybdate reagent.

G. C. J.

Volumetric Estimation of Mercuric Chloride. G. Bressanin. (*Anal. Chim. analyt.*, 1910, **15**, 413-415.)—The method described by Archetti (ANALYST, 1901, **26**, 108) is empirical, and the neutral point of the titration, as shown by phenolphthaleïn, does not correspond with the formation of any definite mercury ammonium compound, or rather, it appears that a number of mercury ammonium complexes are formed. Operating with $\frac{N}{10}$ solutions, it is found that the neutral point to phenolphthaleïn is reached when 8·55 c.c. of $\frac{N}{10}$ ammonia have been added to 10 c.c. of $\frac{N}{10}$ mercuric chloride solution. If, however, an excess of ammonia be added to the mercuric chloride solution, and the excess of ammonia be titrated in an aliquot portion of the clear solution after the precipitate has settled, it will be found that exactly equivalent quantities of ammonia and mercury chloride have been used in the formation of the precipitate. Litmus paper should be used as indicator in the titration, and at least 4 molecules of ammonia (NH_3) must be present for each molecule of mercuric chloride. W. P. S.

Volumetric Determination of Nitric Acid in Nitrocellulose, Nitroglycerol, and other Compounds. E. Brouvert. (*Bull. Soc. Ind. Mulhouse*, 1910, **80**, 254-263.)—The gasometric method of Lunge or of Schloesing is generally employed, or the sample may be distilled, with concentrated sulphuric acid or phosphoric acid, into standard alkali, with subsequent back-titration. The author's method consists in setting free the nitric acid with sulphuric acid, and running in standard stannous sulphate solution from a burette, until the blue coloration produced by diphenylamine is discharged on the reduction of the nitric acid. The solution of nitrocellulose, even of hard powder-grains, is more easily effected by this method than by the others, as the acid may be warmed; any excess of it may be used, and the substance may be crushed, if necessary. The standard tin solution is prepared by dissolving stannous sulphate, or the double sulphate of tin and potassium, in a mixture of equal volumes of sulphuric acid and water, and adding just enough concentrated hydrochloric acid for solution. It is standardised by running it from a burette into a solution made by dissolving 0·15 grain of pure potassium nitrate in a small quantity of water, and adding to it rapidly 50 c.c. of concentrated sulphuric acid, and 1 drop of diphenylamine in water. It is claimed that the method is simpler and more rapid than the others, and at least as accurate. Sample analyses: for a nitrocellulose from cotton, 10·58, 10·60 per cent., against 10·70, 10·60 by Lunge's method; for a wood nitrocellulose, 10·59, 10·56, against 10·20, 10·30 by Lunge's method; for pure potassium nitrate, 99·7, against 98·84 by Lunge's method; for spent acids, 9·73, against 9·7 by Lunge's method; and 9·7 by distillation at 170° C. with phosphoric acid.

O. E. M.

International Standard Units for the Measurement of the Radio-activity of Radium. A. Jaboin. (*J. Pharm. Chim.*, 1910, ii., 494-497.)—Two methods are employed for the valuation of radio-active substances—viz., (1) measurement of the quantity of γ rays emitted by a given weight of the radium, etc., by means of a sensitive electroscope; and (2) measurement of the emanation emitted within a given time from a definite weight of the substance. For purposes of comparison it is necessary to have a standard sample of radium, and it was decided at the recent Congress of Radiology in Brussels that Madame Curie should prepare a specimen of radium, containing about 20 mgrms. of the element, and that this should be kept in Paris as a specimen upon which other preparations could be standardised. It was decided further that the unit of weight should be 1 grm. of radium (element), and that the unit of emanation, to be termed a "curie," should be the quantity of emanation in equilibrium with 1 grm. of radium. By radio-active equilibrium is understood the moment at which a given quantity of radium gives its maximum emanation—*i.e.*, when this quantity of emanation developed in a closed vessel becomes constant. The quantity of emanation given off by a mgrm. of radium (element) is, in like manner, to be termed a "milli-curie," and that emitted by a microgramme of radium (element) a "micro-curie."

C. A. M.

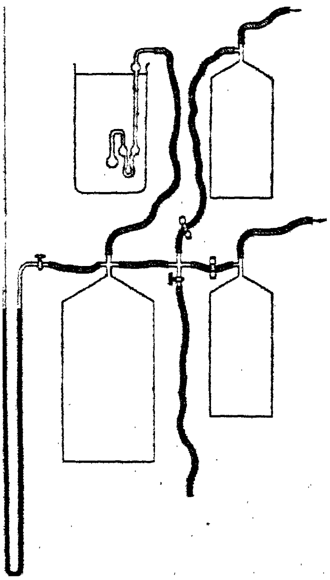
Precipitation of Vanadic Acid as Silver Vanadate, and Estimation of Phosphoric and Vanadic Acids in Presence of each other. G. Edgar. (*Amer. Chem. J.*, 1910, **44**, 467-472.)—In solutions of alkali vanadate containing an excess of carbonate, but no other substance precipitated by silver nitrate, vanadium may be estimated as follows: The solution is acidified with nitric acid, and boiled to expel carbon dioxide. A yellow colour, at first deep and then fading somewhat, is developed by this treatment. Dilute caustic alkali is added to the boiling solution until this yellow colour just disappears. The solution is then faintly alkaline to phenolphthaleïn. Excess of standard silver nitrate is next added, the resulting precipitate, which is of variable composition, is dissolved in ammonia, and the solution then boiled until the odour of ammonia has disappeared. The resulting red precipitate is silver orthovanadate, free from pyrovanadate. After cooling, the precipitate is filtered off and washed, and the excess of silver in the filtrate and washings determined by titration with ammonium thiocyanate, after acidifying with nitric acid. Each c.c. of $\frac{N}{10}$ silver nitrate = 0·00304 grm. V_2O_5. The method as described gives results accurate to 0·05 c.c. $\frac{N}{10}$ silver solution. Under other conditions the silver precipitate may consist wholly of pyrovanadate or be a mixture of ortho- and pyrovanadate.

Phosphoric acid may by precipitated in a similar manner as yellow silver phosphate, Ag_3PO_4, and the following method is recommended for the estimation of phosphoric and vanadic acids in presence of each other. Both acids are precipitated by means of standard silver nitrate exactly as described for vanadic acid alone, and the excess of silver nitrate determined by titration with thiocyanate. The difference between the quantity so found and that taken for the precipitation is the measure of the combined phosphoric and vanadic acids. The vanadic acid is then estimated separately by dissolving the silver precipitate in sulphuric acid, reducing the vanadic acid by means of sulphur dioxide and titration of the lower oxide of vanadium with permanganate in the usual manner. The phosphoric acid is estimated by difference. One c.c. $\frac{N}{20}$ permanganate = 0·00456 grm. V_2O_5 and 1 c.c. $\frac{N}{10}$ silver = 0·00304 grm. V_2O_5 = 0·002367 grm. P_2O_5. The error of the method is no greater than corresponds to 1 drop of the standard solutions employed. G. C. J.

APPARATUS, ETC.

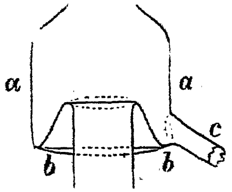
Static Method for Determining Vapour Pressures of Solids and Liquids. A. Smith and A. W. C. Menzies. (*J. Amer. Chem. Soc.*, 1910, **32**, 1412-1434.)—The apparatus is shown diagrammatically in the sketch. The substance, of which about 1 c.c. suffices, is contained in the bulbed tube, termed an isoteniscope, and, if a liquid, acts as its own confining liquid. Owing to the low sp. gr. of most liquids compared with that of mercury, when the levels in both limbs of the U-tube appear to be identical, the error, converted into mercury height, is negligible. The isoteniscope, which is about 24 cm. long over all, is placed in a tall 2-litre beaker containing some suitable liquid, together with a platinum resistance thermometer and a mechanical agitator capable of imparting violent movement to the liquid. Only by violent stirring can thermal equilibrium be obtained. The isoteniscope and the mercury gauge are connected with a large iron bottle. Another piece of tubing leading from this bottle gives connection at will with (1) the atmosphere, (2) a vacuum reservoir and pump, or (3) a pressure reservoir and compression pump.

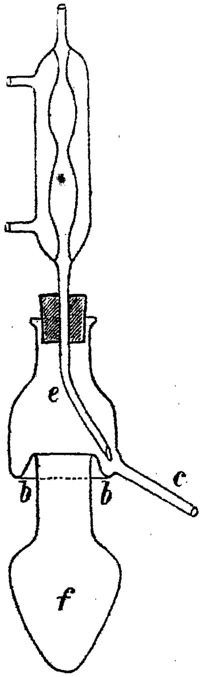
When the temperature of the bath in which the isoteniscope is immersed has reached the desired point and is constant, the exit to the suction bottle and pump is opened and kept open until the substance in the bulb boils, and the vapour passes freely through the confining fluid, which is the liquid phase of the substance itself when a liquid is under examination. (To determine the vapour pressure of a solid, the end bulb of the isotemiscope must be cut off, the solid inserted, and the bulb re-fused to the U-tube. A confining liquid is then chosen, such as has no appreciable vapour pressure of its own at the temperatures to be employed, does not dissolve the vapour of the substance experimented with and does not interact chemically with the vapour.) The confining fluid is driven into the remote side of the U-tube and the bulb above it. When the air in the bulb and the gases adhering to the bulb and dissolved in the liquid have been driven out, air is cautiously admitted to the iron bottle by connecting this with the atmosphere until the levels of the liquid in both limbs of the U-tube are identical. On account of the cooling of the substance by evaporation, equilibrium is not reached at once, so that the adjustment has to be repeated until the levels remain constant. The stopcock on the gauge is then closed, the temperature reading is confirmed, and the gauge and (since this is an open gauge) the barometer are read. The whole process of boiling out, adjusting, and reading is repeated until constant values are obtained. The temperature of the bath is then raised and a new observation taken.



Older methods are criticised, and the several advantages of the present apparatus fully set out. The manipulations involved are incomparably simpler than those of any other static method, and figures are given showing that the method may be made to yield numbers of a high order of accuracy. A series of determinations made with water agree more closely than do those of Regnault, Magnus, Battelli, or Wiebe with the smoothed values of Holborn and Henning (*Ann. Physik.*, 1908, [iv.] 26, 833), which are undoubtedly the most accurate in existence, and the comparison was made, not with smoothed values obtained by graphical or other methods, but with individual results, subject to the irregularities which individual observations usually show. G. C. J.

Microdistillation Apparatus. A. Gawalowski. (*Zeitsch. anal. Chem.*, 1910, 49, 744-745.)—The left-hand drawing illustrates, half-size, a glass vessel in one piece for the distillation of small quantities of liquid. It is closed at the top [illegible] by a glass ball, a narrow-necked funnel, or a cork carrying a condenser, and the distillate flows away at *c*. The right-hand drawing represents (one-third natural size) a similar





apparatus, with the difference that the lower part, instead of being a simple tube, is given a shape which militates against frothing. G. C. J.

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REVIEWS.

The Extra Pharmacopœia. By W. H. Martindale, Ph.D., and W. Wynne Westcott, M.B. H. K. Lewis. Pp. 1054. Price 12s.

The fourteenth edition of this well-known work appears in a new and condensed form; and now, although much fresh matter has been added, the contents have been compressed, without any loss of clearness, into a volume of a little more than half the original size.

The scope of the book is sufficiently familiar to most. It appeals mainly to the physician and the pharmacist; but there is much matter relating to analytical work, and there are many paragraphs dealing with topics somewhat off the beaten track on which the practising analyst may find information, the value of which is increased by references to original papers.

The main bulk of the work is taken up with an account of the properties and uses of medicinal substances, the ground covered being similar to that dealt with in Squire's "Companion to the British Pharmacopœia"; but, in addition to this, there are pages or paragraphs dealing with the following: Analysis of urine, blood, stomach contents, water (chemical and bacteriological), milk, butter; tests for carbon monoxide poisoning; bacteriological notes dealing with the characters and methods of cultivating and staining the chief organisms associated with specific diseases; the preparation of culture media; embalming; and the antiseptic power of chemical substances. There are also notes on the Poisons and Pharmacy Acts, a list of scheduled poisons, and a glossary in six European languages of words and phrases likely to be met with in prescriptions.

Amongst the new subjects dealt with are lactic acid bacilli and curdled milk preparations and methods for their examination, organic arsenic compounds, the introduction of drugs into the system by electrical means (iontophoresis), and the use of radium and its preparations in medicine. A list of some two or three hundred proprietary medicines, giving the chief active ingredients stated to have been found in them, is the more valuable because the source from which the information is derived is in almost every case given.

It will thus be seen that there is a vast mass of information with much of which the practising analyst has little direct concern, but a good deal of which he might on occasion find of use. The extremely wide scope is the *raison d'être* of the book, and at the same time one of its weakest points, so far as the inclusion of analytical matters is concerned.

Condensation has obviously had to be carried to its extreme limits, and, although the style is admirably clear (though not always elegant), it is quite impossible, in such brief space as could be afforded to any single subject, to point out the danger that may lurk in even the simplest analytical process when in inexperienced hands, such as those of the majority of "medical practitioners and students," for whom the analytical notes are said in the Preface to be mainly intended. When, for instance, the whole art and science of water analysis is dealt with in the space of a page and a half, including the preparation of the standard solutions and notes on the interpretation of results, the inexperienced person, with no other book at hand, is naturally led to believe that the subject is a very simple one. How far he might be led astray will be obvious when it is mentioned that only a qualitative test for nitrates is given—viz., the brucine reaction.

It is fair to add that such serious omissions are the exception, and that the book must be allowed the defects of its qualities. Moreover, it is as a general book of reference, and not as a manual of analytical chemistry, that it will appeal to the readers of this journal.

Regarded simply as universal providers of medico-chemical learning, it must be admitted that the authors have done their work well, and few books of this kind contain a greater volume of useful information to the cubic inch.

CECIL H. CRIBB.

ORGANIC ANALYSIS CHART. By W. HARRISON MARTINDALE, Ph.D. H. K. Lewis. Pp. 80. Price 3s. 6d.

This is a supplement to the above work (p. 39), intended to assist in the recognition of a number of organic chemicals used in medicine.

The first sixty-three pages are devoted to a series of tables, which give in the briefest possible manner the result of certain chemical and physical tests on the selected substances.

In addition to tests involving the use of certain specified reagents, the action of heat, the specific gravity, the melting-point and the solubility or miscibility with water or alcohol, are also given. The reagents include acids, alkalis, Fehling's solution, and certain general alkaloidal reagents, and the substances about which the information is given—319 in all—include a very large number of bodies not at all commonly met with, such, for instance, as enesol, ephedrine, hetraline, largin, neurodin, and tylmarin. It is, however, rather difficult to account for the selection made by the author, because, along with the out-of-the-way substances just mentioned, such common things as cane sugar, lævulose, and dextrose are also referred to, and yet others equally common — *e.g.*, glycerol, maltose, and lactose — are missed out.

It is obvious that identification is hardly likely to be arrived at in this way, but to render this possible the final fourteen pages of the book are devoted to corroborative tests, one or more being given for most of the substances mentioned in the tables, or else references are given to larger works.

The idea is a practical one, and as it is applied to many bodies about which little information is available, it is likely to be useful to those confronted with the problems with which it is meant to deal. CECIL H. CRIBB.

LA CHIMICA DELLE SOSTANZE ALIMENTARI. P. E. ALLESANDRI. Published by U. Hoepli, Milan. Price 6 lire, 50.

Although this book is only a small octavo volume of 600 pages, the author has endeavoured to compress within these limits an enormous quantity of material; for it includes not only full descriptive methods for the analysis of water, wine, milk, and food products of every description, but also contains sections upon air, petroleum, coal-gas, perfumes, and cosmetics.

The section dealing with water occupies about a quarter of the available space, and gives a most valuable outline of the methods of examination and of the various points upon which a judgment of the results should be based. It includes illustrations of the unicellular and amœboid organisms of most common occurrence in water.

The section on wine is also excellent, and includes a description of the various maladies to which wine is subject; but in most of the other parts of the book the evils of compression are obvious, and in many instances the methods are not up to date. Thus, of the twenty pages devoted to oils, a good deal of the space is occupied by descriptions and tables of special hydrometers and of obsolete colour reactions with phuric acid, etc., while no reference is made to Wijs' method of determining the ine value.

In the section upon butter the reader is directed to take 2·5 grms. of fat for the ermination of the Reichert-Meissl value, and the author is evidently under the)ression that "Reichert-Meissl" was the compound name of one man, whose thod was subsequently modified by Wollny.

Points such as these, which might be largely multiplied, are sufficient to show t the book is of unequal merit, and does not reach the standard of its first portion. most of the sections useful facts and methods may be gleaned, but the reader will many cases have need to refer to other books upon the analysis of food.

C. A. MITCHELL.

E SPARK SPECTRA OF THE METALS. By Admiral GEORGE E. GISSING. 1910. London: Baillière, Tindall and Cox. Demy 4to. Price 7s. 6d. net.

is book the author has described the spark spectra of all the metals, with f aiding those engaged in the qualitative investigation of alloys or minerals. of all the spectra are quite uniform, and will serve as very handy references rison when any substance is examined spectroscopically with a view of ng its metallic constituents. Only the visible region is dealt with, but the park spectra are sufficiently characteristic over this range for the particular n view. Among the spark spectra are included the vacuum-tube spectra of elium, hydrogen, and carbon dioxide. In the text are given careful ions for the use of those who are novices in this particular branch of work. are also added notes of the most important and characteristic lines of each of ectra illustrated. If any criticism were needed, [illegible] might perhaps be said that thor is unduly severe when he remarks that spectrum analysis is of no use at quantitative analysis. He is hardly fair to his own subject in saying this, it would certainly seem that we have in our hands, as the result of Pollok's d that of Count de Gramont, the basis of an accurate quantitative method. course, this little book, with its fifty reproductions of spectra, good as they are, ank among the well-known spectrographic atlases. This, however, may be as a virtue, for it is far easier to recognise a spectrum by comparison with tographs as those of Admiral Gissing rather than with the big maps given atlases.

E. C. C. BALY.

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NEW BOOK.

THE SCIENTIST'S REFERENCE BOOK AND DIARY FOR 1911. For James Woolley, Sc and Co., Ltd., Manchester. By CHAS. LETTS and Co. London. 1911. P 1s. 6d.

Part I. contains lists of Associations, Institutes, Congresses, International Ex bition (Turin), etc., Scientific Societies, Data as to First Aid in poisoning ca A calendar, and a "daily wants" dictionary then follows, also a series of blank pa for memoranda, telephone numbers, etc. A Diary for 1911, and blank pages addresses, complete the part. Part II. is back to back with this, as a duplic pocket-book, and forming the "Scientist's Reference Book," with numerous ite such as Astronomy; Meteorology; British Weights and Measures; Metric Weig and Measures, etc.; Chemical Terms, Synonyms, etc.; Atomic Weights; Speci Gravities; Physical Constants of the Elements; Formulæ and Solubili Water; Factors for Use in Volumetric Analysis, etc.; Alloys; Scientific Te in general use; Laws and Definitions in Chemistry and Physics; also Human Physiology; Hygiene; Chemical Composition of Food. A serie blank pages completes the booklet.

FEBRUARY, 1911. Vol. XXXVI., No. 417

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ANNUAL ADDRESS OF THE PRESIDENT.

(*Delivered at the Annual General Meeting, February* 1, 1911.)

In presenting my annual address, it is my first duty to refer to the losses we have sustained during the session. The year 1910 was shadowed by the tragically sudden and deeply mourned death of a Sovereign who had so firmly attached to himself the affections, not only of his own peoples, but those of foreign nations also. Your Council presented a loyal and dutiful address of sympathy to His Majesty King George V., and this address was graciously received and acknowledged.

Of the members of our Society, two have passed away, both of them men of mark. One, Dr. J. Campbell Brown, was an original member of our Society, and Public Analyst for Lancashire. The second member whose loss we deplore was Oscar Guttmann, the well-known expert on explosives, who was a comparatively recent member of our Society. Four others have ceased to continue their membership; but we have had, on the other hand, an addition of twenty-one new members, including one honorary member, Dr. J. J. Dobbie, Principal of the Government Laboratory, whose accession is a source of great satisfaction and pleasure to us.

Our total membership now amounts to 380, and we are in the healthy condition of having a much heavier birth-rate than death-rate. The increase in our membership is, however, not as large as I could wish, although it is very encouraging, especially from the point of view of the *personnel* of our new members.

I cannot help feeling that there are many of our professional brethren whose inclusion in our ranks would very materially add to our usefulness, and I feel sure that if members would make a personal effort, an increased membership would be obtained. We carefully avoid holding out any pretensions that we, as a Society, are a "qualifying body" in any way; but, at the same time, it is imperative to the well-being of our profession that we should strictly uphold the dignity of our Society, and we are entitled to insist that anyone proposed as a member should be scientifically and socially qualified. I would, therefore, urge members to exercise the greatest care in the matter of signing proposal forms. It is undoubtedly often difficult and unpleasant to refuse to do so; but I would remind members that, in affixing their signature to a form, they are accepting a personal responsibility that the candidate is strictly complying with the requirements laid down in our Articles of Association.

That membership of our Society does confer a distinction, I would be the last one to question, but I very strongly deprecate any attempt to make use of the membership of this Society as a qualification, and I would appeal to members to avoid any appearance of this. One instance occurs to me, and one which I think we must all condemn, where a gentleman, who resigned his membership many years ago, still describes himself, when he appears in print, as a "Past Vice-President" of our Society. In connection with the increase of our membership, it has been suggested that possibly a resumption of those country meetings, which used to be held in former days, would materially assist in creating a greater interest in the Society among provincial analytical chemists. The matter is one which I would suggest might be carefully considered by the Council.

The financial statement which our Treasurer has laid before you is, I venture to say, extremely satisfactory; and although the available balance at the end of the year is less than it was a year ago, it must be noted that there are items of extraordinary expenditure, which largely account for this—for example, our contribution of fifty guineas to the Building Fund of the Institute of Chemistry. There is also the item of expenses in connection with the visit of our delegate to the International Conference held at Paris, to which I shall subsequently refer.

Our Treasurer is making timely provision for a serious expenditure which we shall in a few years have to face in connection with a new General Index of the ANALYST. The Society may congratulate themselves on having secured the services of a Treasurer who is not merely careful of our present resources, but who looks forward to proper provision for future expenditure. You will observe that he has taken a step which I am sorry that I failed to do when I held the like office. I allude to the exchange of our holding in Government securities to that in Colonial securities, yielding a better return of interest, and having practically equal security. Consols, as you are aware, have persistently declined in value, and we have had more than once to seriously write them down in our books. It is to be noted that, since the change effected by Mr. Richmond came into effect, the Consols disposed of have fallen in value, while the newly-purchased stocks have appreciated.

Our Journal, the ANALYST, continues year by year to increase, not only in substance, but in quality, and it is now more and more widely recognised as the most valuable Journal of analytical chemistry. Our most cordial thanks are due to the Editor, Mr. Julian L. Baker, for the able manner in which he conducts our Journal, and for the care which he exercises in securing abstracts of all important papers appearing in foreign and other scientific journals. During each of the last two years nearly seven hundred abstracts have appeared in our Journal, while four years ago, when Mr. Baker first took over the editorship, only about four hundred abstracts appeared.

The work under our analytical research scheme continues to flourish, and so far thirty-four investigations have been undertaken, the results of many of which have been published in the ANALYST. During 1910 nine investigations were commenced, five of which have been completed, and the results published during the year; thirteen are not yet completed, and arrangements have been made for several new subjects to be taken in hand. I consider that this research scheme is doing most useful and valuable work, and is one which ought to be more actively supported than it is.

You will notice in the Treasurer's statement that there is a small deficit in the Research Fund, the income of which has not been sufficient to meet the calls upon it. As you are aware, the scheme was suggested by our Hon. Secretary, Mr. Chapman, and the fund was started by the members of the Publication Committee, and no definite appeal for funds has been made to the general body of our members. The time has now arrived when I think such an appeal might be made, and I venture to do so in the hope that many of our members who recognise —as they must—the enormous importance and value of the work which is carried out under this scheme will generously support it, and put it in such a position that the income derived from its Capital Fund will be sufficient to meet the calls upon it.

At the seven meetings held during the past session, thirty-six papers were communicated to the Society, and as the records of these appear in our Journal, I depart from the usual practice of my predecessors of giving the list of these papers. It is sufficient to say that many of them have been of exceptional interest, and that they have dealt with a very wide range of subjects.

One of the great disadvantages of our scientific journals of the present day is the amount of overlapping which takes place. Abstracts of papers are found dotted about here and there, and appear over and over again in different journals, thus involving a great waste of time. It would be a great advantage to the scientific world if a serious effort could be made to concentrate this work, and to avoid this overlapping. Not only is this the case with abstracts, but papers of a purely analytical interest are communicated to societies which are not really directly concerned in the subject. I would appeal to members to forgo the natural temptation to come before a larger public than our own society can command, and to remember that our meetings are those to which such papers ought to be communicated.

Early in June we received, at the instance of the Foreign Office and of the Local Government Board, an invitation to send a representative to an International Conference which was to be held in Paris at the end of that month. This Conference had been summoned by the French Government to discuss two questions—first, that of the Unification of Methods of expressing the Analytical Results obtained in the Analysis of Food Products; and secondly, that of the Unification of the Methods of Analysis.

The British Government had not been officially represented at previous International Conferences on these and kindred subjects, and the Foreign Office thought it desirable that on this occasion this country should be officially represented.

On receipt of the invitation from the Local Government Board, the Council of the Society delegated our Hon. Secretary, Mr. Chapman, as their representative. The other British delegates to the Conference were: Dr. G. S. Buchanan and Dr. G. W. Monier-Williams, of the Local Government Board; Dr. J. J. Dobbie, Director of the Government Laboratories; Mr. E. J. Connah, Chief Chemist of the Customs Laboratory; and Dr. Nestor Tirard, Secretary of the Pharmacopœia Committee of the General Medical Council.

The following countries were also represented: Argentina, Belgium, Bulgaria, China, Denmark, Greece, Holland, Hungary, Italy, Mexico, Norway, Portugal, Sweden, Switzerland, and Uruguay, in addition to the French delegates. Dr. Bordas, the Principal of the Laboratories of the French Ministry of Finance, was appointed President. The first four meetings of the Conference were occupied with the discussion of the Unification of the Methods of Expressing Analytical Results, a matter on which it would appear that it should be possible and advantageous to come to an International agreement. The French delegates had prepared a statement of their proposals which served as a basis for deliberation. Mr. Chapman, in consultation with his colleagues on our Council, had prepared an extremely careful and able memorandum on the subject, and it is a matter of congratulation to know that his suggestions not only had the approval of the British delegates, but also that many of them were adopted by the Conference. As a result, the final report on this portion of the Conference was signed by all the British delegates who, however, were obliged to make it clear that, however strongly they might feel as to the desirability and advantage likely to result from the adoption of the recommendations, they could not be answerable for their adoption in Great Britain.

The question of the unification of methods of analysis was one on which there was little probability of any sort of agreement, and the matter was only discussed in general terms at the last two meetings of the Conference. It was, however, finally resolved that the French Government should consider a scheme for the creation of a permanent International Bureau, with laboratories, where the study of methods of the analysis of food products, with a view of their possible unification, should be carried out, and that this scheme should be submitted as soon as possible to all the Governments represented at the Conference. It is a matter to be noted, in connection with this International Conference, that Germany, a country particularly addicted to "Conferences," did not send any delegates.

Mr. Chapman desires to gratefully acknowledge the very courteous and kindly manner in which he, as the Society's representative, was received by the French authorities.

In October the Second International Congress of Food Hygiene met at Brussels, the Congress being divided into seven sections, when a programme embracing Physiology, Physiological Chemistry, Hygiene, Bacteriology, the Analysis and Adulteration of Foods, Legislation for the Repression of Frauds, Water Supplies, and Alcoholic Liquors was put forward.

The report of the proceedings of this Congress has not yet been published, but it is difficult to believe that a programme so ambitious, and covering so wide a field of inquiry, could possibly be adequately dealt with in the four days allotted to it. I must confess that I am very sceptical as to the practical value of these International Congresses, and I doubt whether they result in any definite progress being made. Indeed, conclusions may be arrived at completely subversive of progress, as was the case at the International Food Congress held in Paris in 1909, when it was decreed that boracic preservatives were absolutely necessary in the manufacture of butter, and that the "standard water-content" of butter should be "raised from 16 to 18 per cent." We had appointed delegates to this Congress, but, as they were unable to be present, we had to be content with publicly protesting against the decisions arrived at.

There has been, during the past year, a considerable agitation with the view of directing attention to the administration both of the Sale of Food and Drugs Act, and of the Fertilisers and Feeding Stuffs Acts. In March a Conference, organised by the County Councils' Association, was held in London, at which, although we, as a Society, were not officially represented, several of our members holding appointments under these Acts attended at the request of the County Councils. A number of resolutions were passed at this Conference, to some of which I will now briefly refer.

With regard to the Fertilisers and Feeding Stuffs Acts, a resolution was passed to the effect that, in cases where samples are taken by an official sampler, "the ten days' limit should be extended to twenty-one days." It was also resolved that it was a matter of urgency that the question of "standards" under the Fertilisers and Feeding Stuffs Acts should be dealt with by an expert Board of Reference.

Under the Sale of Food and Drugs Acts it was recommended, (1) That it would be desirable if wholesale manufacturers could be brought within the provisions of the Food and Drugs Acts with regard to the taking of samples; (2) that the powers of vendors to contract themselves out of the Acts by means of a notice should be severely restricted; (3) that the British Pharmacopœia should be made a legal standard for medicinal articles, but not until the new edition was compiled by the General Medical Council. On the question of "standards and definitions," it was resolved that the Local Government Board should have power to fix standards and definitions of purity for articles of food, and to order the nature and amount of preservative or other ingredient in any article of food to be declared on the label. It was also resolved that the Board of Agriculture should have power to fix "standards" for milk, cream, butter, or cheese, which should be conclusive evidence as to their not being genuine, and not merely raise a presumption until the contrary were proved. They also recommend that an expert Board of Reference should be constituted to decide controversial questions of chemistry, hygiene, or physiology.

The questions of standards and definitions was referred to two of the Public Analysts who were present at the meeting, Mr. Hinks and Mr. Duncan, with a view to obtaining the opinion of representative analysts as to the framing of a list of articles for which it would be advantageous that there should be official fixed standards and definitions of purity. These gentlemen reported that the result of their inquiries did not justify them in making specific recommendations, and that the consideration of these questions could only be taken in hand in detail by a representative body such as the Society of Public Analysts or by an expert Board of Reference.

The President of the Local Government Board subsequently received a deputation of the County Councils' Association, and expressed himself as being in complete sympathy with their objects. He pointed out the difficulty of introducing fresh legislation on the subject, and the enormous amount of opposition any amendment of the Acts would give rise to. He suggested that a simple Bill, giving the Local Government Board the power of determining questions of trade practices, and enabling it to issue regulations which would have statutory effect, would be a possible solution of the difficulty, and informed the deputation that he would arrange that some of the staff of his Department should confer with members of the deputation to see whether it would be possible to introduce legislation to meet the case.

The question of any amendment of the Sale of Food and Drugs Acts is one in which we as a Society are profoundly interested, and I venture to say that our views, as representing the Public Analysts of the kingdom, must have the most prominent and careful consideration if any real advance is to be made.

The difficulty of getting Parliament to move in the matter is well known. There are, no doubt, advantages in engineering a popular crusade against adulteration. The danger, however, lies in the tendency of all such movements to run riot in the effort to attract public attention and interest. As an example of this I would remind you of what took place last year at the Pure Food Exhibition, when most outrageous statements were made in the public Press as to the manner and extent to which different foods, as supplied to the public, are adulterated.

There is no question that there is a crying need for revision of our Food Acts, and to strengthen them in such a way as will enable the widespread and skilful tampering with our food products to be checked. It is, I think, most unfortunate that, when the amended Act of 1899 was introduced, the most important recommendation of the Select Committee of the House of Commons on Food Products Adulteration (which reported to Parliament in 1896) was not acted upon. I refer to the formation of a Court of Reference, which should have power to decide as to what does or does not constitute adulteration, and which would be competent to deal with the question as to whether the many trade practices which have grown up in connection with the manufacture and preparation of so many of our foodstuffs are legitimate or otherwise.

With all endeavours in the direction of bringing pressure to bear on the authorities responsible for the administration of the laws against adulteration, we as a Society have the most cordial sympathy; but, at the same time, we recognise that the whole subject bristles with difficulties, and requires handling with the greatest care, and by people who are thoroughly conversant with all sides of the question. The protection of the public against adulteration is a function of the State, and cannot be delegated to any self-constituted body, no matter how good and disinterested the motives of that body may be. I think it is necessary to point out to Public Analysts that their duty lies solely in the direction of the detection and prevention of fraud.

It has been urged sometimes that it is not fair that the vendor of any article coming under the Food and Drugs Acts should be held liable for any occasional fault which can be discovered, while he is prevented from utilising the benefits which would accrue to him as a trader were equal prominence given to the satisfactory reports received upon his products. I maintain most strongly that it is no part of the duty of the Public Analyst to push the business of trade rivals in this way, and that it is especially incumbent upon members of our profession who hold official public appointments to avoid giving reports to traders for the purpose of advertising their wares.

The suggestion that official standards should be laid down for all articles coming under the Food and Drugs Acts and the Fertilisers and Feeding Stuffs Acts is one which appeals to many as the simplest and most satisfactory solution of the difficulty; but, apart from the practical impossibility of definitely fixing the composition of food products which naturally show very wide variations, there is in some cases the objection that the fixing of such standards will inevitably tend towards the lowering of the general quality of the article. That this has actually taken place is strikingly shown in the case of butter. The fixing of a maximum limit for the percentage of water has undoubtedly resulted in an enormous increase in the number of samples which are found to contain an amount of water closely approximating to this maximum.

During the past year there have been, so far as I am aware, no High Court decisions in food and drugs cases which have had an important bearing upon the carrying out of the Acts.

In the inferior courts, however, several cases have occurred which strikingly show the necessity of an authoritative tribunal which will enable courts to decide whether a particular practice of a trade is legitimate or otherwise. It is satisfactory to note that a magisterial decision has been given to the effect that "golden syrup" is a definite article, and is understood by the public to be "refined treacle." Convictions for selling as "golden syrup" a syrup containing large admixtures of glucose syrup have been obtained, in spite of the contention that such syrups were "improved" commercial products.

The cases dealing with the question of what is cocoa are of special interest. Public Analysts who have been investigating this matter very thoroughly found that there was an increasing trade practice of mixing the ground shell, removed in the manufacture of high-class cocoa, with much of the other cocoa sold to the public. Several prosecutions were instituted in cases where the samples of cocoa were certified to contain from 35 to 60 per cent. of cocoa-shell, and the magistrate, after hearing evidence to the effect that the maximum amount of shell which cocoa would naturally contain if none of the shell were removed in the manufacture was about 12 per cent., decided that cocoa containing added cocoa-shell was not cocoa, and convicted the vendors. This decision, however, has not long remained unchallenged, for in a recent prosecution for the sale of cocoa containing 18 per cent. of added shell, the same magistrate, after hearing much conflicting evidence from experts and manufacturers, dismissed the summons.

It must be welcomed as a significant sign of progress that during the term of office of the present President of the Local Government Board a special Food Department has been organised, under the direction of Dr. Buchanan as Chief Inspector of Foods.

Dr. Buchanan, in addition to receiving the assistance of several Medical Inspectors, has now been able to add to the equipment of the Board a well-appointed chemical laboratory, and it is gratifying to state that a member of our profession and of our Society, Dr. Monier-Williams, has been chosen to co-operate with Dr. Buchanan in the direction of this laboratory.

Several most useful and important investigations have been undertaken, and reports dealing with the facing of rice, the use of preservatives in cream, and the various methods of preserving meats imported into this country, have been issued, and others are in course of preparation.

One cannot help feeling that, if some such step had been taken thirty years ago, it would have been of immense advantage; but we must, at any rate, be glad that so necessary a step has at length been taken.

The position of the Public Analyst is one which gives rise to a feeling of considerable anxiety as regards the future. The responsibility and the amount of work devolving on him increases year by year, and he has to be constantly on the alert in order to be able to cope with new and subtle forms of food sophistication. It cannot be denied that the Public Analyst has never had that recognition which he ought to have had. This I attribute in great measure to the indifference of the State to science in general and to chemistry in particular, which has so long prevailed.

When the Sale of Food and Drugs Act was first brought in, the science of food analysis was in its infancy, and the number of men really competent to undertake the responsible work of Public Analysts was extremely limited. The department, moreover, responsible for the Act—the Local Government Board—was guided, and always has been, in great measure, by medical authority, and, in controlling the appointments under the Act, has too often in the past failed to recognise the all-importance of chemical qualifications. Indeed, I believe that their former ideal of a Public Analyst was a medical man who had acquired some chemical training.

We are fully alive to the fact that there are, and have been, medical men with chemical qualifications of the highest order, who have rendered most valuable service to the profession of analytical chemistry, but it must be admitted that such men are the brilliant exceptions, and that the training in chemistry of the average medical man—even when supplemented by special work in connection with Public Health diplomas—cannot be regarded as in any way sufficient to qualify him for carrying out the duties of a Public Analyst.

We gratefully recognise that a very great change has been effected in the views held by the Local Government Board as to the importance of a proper chemical training such as is insisted upon by the Institute of Chemistry, and that now the diploma of this body is treated as a necessary qualification. The attitude of the Local Government Board in the past has naturally tended to shape that of local authorities, and the office of Public Analyst is too frequently regarded as an altogether inferior department of the Medical Officer of Health.

The Local Government Board has shown itself to be in accord with the efforts of the Institute of Chemistry and of our own Society to improve the status of the Public Analyst, and by their refusal to confirm appointments of men who have not the requisite chemical training, and by their clearly expressed disapproval of the practice of many local authorities of offering the position of Public Analyst to competitive tender, have done much to remove serious grievances. The Board, however, have not the control over local authorities which it is desirable they should have, and the Public Analyst has not that security of tenure of office which it is not only in his interest, but in that of the public, which he should have.

In the matter of the remuneration of the Public Analyst, the Board profess themselves unable to interfere, even when the remuneration is, in their opinion, utterly inadequate. The difficulty of this question is aggravated by the fact that so many analysts have, in their anxiety to secure positions, indulged in a system of wholesale undercutting of fees. Local authorities cannot be blamed if, regarding—as they naturally do—the question of economy in the administration of the Acts as one of the most important points, they take advantage of the willingness of properly qualified men to work under unremunerative terms. Recent cases which have occurred in Ireland show to what a deplorable condition things may be brought by this altogether unprofessional competition to secure appointments.

The proper training of a Public Analyst involves a long and expensive course of study and practice, and if in future the professional emoluments of Public Analysts is to be placed on a low scale, it is difficult to believe that the rising generation of chemists will be willing to undergo the severe course of training required of them.

There is no use shutting our eyes to the fact that the obvious tendency of the present day is for local authorities to make the Public Analyst a "whole time" officer, and with this it would appear that the Local Government Board is in complete sympathy. In a Memorandum recently issued by the Board, there was a strong recommendation to local authorities that Medical Officers of Health should not be engaged in private practice, and that appointments should be arranged so as to secure the "whole time" services of this officer. Special stress, however, was laid on the necessity of local authorities offering adequate remuneration in order that the services of well qualified men might be secured and retained. One could wish that similar directions as to remuneration could be given in the case of the appointments of Public Analysts, for it would be a misfortune if these appointments were in the future to be so inadequately paid as to fail to attract the services of men of standing in the profession.

Personally, I cannot help feeling that there are decided disadvantages from the point of view of the public interest in placing the analyst in a position of complete dependence upon local authorities.

Another point which I think has acted detrimentally to our interests is the lamentable ignorance of the public generally as to the proper meaning and function of a "chemist." Unfortunately, the title of "chemist" is, by Act of Parliament, ear-marked as belonging to pharmacy, and a large proportion of the public consider that the profession of analytical chemistry and the compounding and sale of medicines are occupations properly carried on by one and the same person. This idea is not confined to the man in the street, who may be excused for ignorance, but is shared in by intelligent people who ought to know better. I have a painful recollection of the remark made by one of the most learned Lord Justices—on the occasion of one of the dinners of the Institute of Chemistry at which he was an honoured guest—who, in replying to a toast, clearly showed that he confused the functions of an analytical chemist with those of the druggist.

When the legislation was introduced which gave rise to the term "chemist" and "druggist," the professional chemist, as we understand him, may be said not to have existed, but I think the time has now come when it would be very desirable if some steps could be taken to correct this confusion, and that the term "chemist" should be confined to its proper meaning.

In concluding my address, I must express my most heartfelt appreciation of the honour that has been conferred upon me, and the fact that the Society has seen fit to do this in the case of two members of the same family, confers on that family a unique distinction. To my colleagues on the Council, and especially to our secretaries, I would wish to express my most grateful thanks for the assistance they have so continuously given me during my term of office.

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WALDEMAR FISCHER'S MODIFICATION OF VOLHARD'S METHOD FOR THE VOLUMETRIC ESTIMATION OF MANGANESE, AND ITS COMPARISON WITH OTHER WELL-KNOWN METHODS.*

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(*Read at the Meeting, December* 7, 1910.)

An examination has been made of the modification recommended by W. Fischer (*Zeitsch. Anal. Chem.*, 1909, **48**, 751; Analyst, 1910, **35**, 80) of Volhard's well-known method for the determination of manganese, with the object of determining its degree of accuracy and of applying it to the estimation of manganese in high-grade ores and alloys. A detailed account of the results is recorded below, from which it will be seen that the process compares very favourably with several other well-established methods.

1. Examination of Fischer's Modification of Volhard's Method.

Volhard's method (*Annalen*, 1879, **198**, 318) for the estimation of manganese consists in titrating a hot solution of manganous sulphate containing zinc sulphate with potassium permanganate, when manganese dioxide is precipitated, a reaction occurring which is essentially represented by the equation

$$3MnSO_4 + 2KMnO_4 + 2H_2O = 5MnO_2 + K_2SO_4 + 2H_2SO_4.$$

The end of the titration is recognised by the supernatant liquid acquiring a faint permanent pink coloration.

Few analytical methods have been subjected to more criticism and modification than that just outlined, and the numerous though frequently conflicting statements concerning it that have appeared from time to time at least indicate that the method is one that appeals to the practical chemist. It has been generally recognised that the method gives low results, which is not surprising when the extreme difficulty of precipitating manganese exclusively in the form of dioxide is remembered (*cf.* Wright and Menke, *J. Chem. Soc.*, 1880, 37, 22; Gorgeu, *Bull. Soc. Chim.*, 1893, **11** [iii.], 490; Gooch and Austin, *Amer. J. Sci.*, 1898, **5**, 260). The presence of chloride is generally held to lower the results still further. An often-used modification of Volhard's procedure consists in titrating in the presence of suspended zinc oxide or calcium carbonate, which neutralises the acid formed during the titration. The temperature of titration also influences the results. It will therefore be apparent that accurate results cannot be expected from any of the usual modifications of Volhard's method unless the working value of the permanganate solution is empirically fixed by titration against a known amount of pure manganese salt.

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The procedure recommended by Fischer (*loc. cit.*) claims to yield accurate results even in the presence of chloride in high concentration, while the permanganate may be standardised with sodium oxalate; it is said that the precipitated manganese dioxide contains the theoretical amount of available oxygen. The following account of the method is quoted from the ANALYST (*loc. cit.*):

"The solution of manganese in hydrochloric or sulphuric acid is diluted to 500 c.c., neutralised with caustic soda until a slight precipitate persists on shaking, and the precipitate just redissolved by cautious addition of dilute sulphuric acid. One grm. of freshly ignited zinc oxide and 10 grms. of zinc sulphate are next added, the solution heated to boiling, and titrated hot with permanganate. When the colour of the permanganate is no longer discharged, 1 c.c. of glacial acetic acid is added. The colour usually disappears, and the precipitate settles instantly. Permanganate is then added drop by drop until the solution becomes permanently pink, when the total consumption of permanganate accurately measures the manganese present."

To the above description it should be added that Fischer directs that the titration should be performed with frequent boiling and shaking, and that in the absence of chloride the quantity of zinc sulphate should be increased (about 10 grms. for each 10 c.c. $\frac{N}{10}$ $KMnO_4$ required).

From the equation given above it will be seen that 1 c.c. of $\frac{N}{10}$* permanganate only corresponds to 0·001648 grm. of manganese present as manganous salt; and, consequently, an error of 0·2 c.c. only corresponds to 0·00033 grm. of manganese. Nevertheless, this means an error of 0·6 per cent. in a titration requiring 33 c.c. The relatively large percentage error corresponding to an actual error of a fraction of a milligram of manganese does not appear to have been properly appreciated by several other workers (*cf.* von Longi and Camilla, *Gazz.*, 1897, **27**, 97), who have reported favourably of procedures which lead to actual errors of several times the magnitude mentioned above.

Materials Employed—(*a*) *Potassium Permanganate.*—The purest commercial salt was twice recrystallised from water at 65° to 70° C., and the product dried over sulphuric acid.

(*b*) (*c*) (*d*) *Manganous Sulphate, Sodium Chloride, Zinc Sulphate.*—The purest commercial preparations were used. Filtered solutions of the two latter salts of known concentration were measured out as required.

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* By *decinormal* permanganate is here meant the usual solution, containing 3·16 grms. of the salt per litre, which is decinormal in *acid* solution.

(*e*) *Zinc Oxide.*—Pure precipitated zinc oxide was calcined in a muffle furnace. It was tested for impurity by de Koninck's method (*J. Soc. Chem. Ind.*, 1905, **24**, 152), with negative results.

(*f*) *Glacial Acetic Acid.*—Kahlbaum's purest preparation was usually employed, but the best commercial acid was found to contain nothing oxidisable by permanganate.

The majority of the test analyses were carried out by the following method, which was used in a series of experiments by Fischer: An approximately $\frac{N}{10}$ permanganate solution was prepared, and portions of 50 c.c. accurately measured out and evaporated to dryness with concentrated hydrochloric acid. The resulting manganous chloride was dissolved in water, rinsed into a litre flask, the requisite amounts of sodium chloride and zinc sulphate added, and the solution titrated by Fischer's process with the remaining permanganate solution. The theoretical volume of the latter required is 33·33 c.c., as will be seen at once from the equation, and three times the actual titration error in c.c. gives the percentage error.

It was originally intended to observe accurately the end-point obtained before the introduction of the acetic acid, and then to finish the titration in acetic acid solution; but it was soon found that this would consume considerable time without yielding results of any practical value. The accurate observation of the first end-point was therefore given up; the titration was carried out in the presence of zinc oxide until within about 1 c.c. of the theoretical value, when the acetic acid was added and the titration accurately finished.

As the result of a large number of test experiments, which frequently led to most curiously inconsistent results, it was eventually concluded that the titration cannot be completed in boiling solution, as directed by Fischer, since under these circumstances it is not possible to rely upon obtaining a definite end-point. Subsequently, it was discovered that this point had been insisted upon by von Longi and Camilla (*loc. cit.*) in their interesting review of Volhard's method. The disappearance of the permanganate end-point in boiling solution is probably accelerated by contact with the manganese precipitate, in view of the well-known instability of permanganate in the presence of the solid dioxide (Morse, Hopkins and Walker, *Amer. Chem. J.*, 1896, **18**, 401).

The following method, differing very slightly in detail from Fischer's, can be relied upon to afford satisfactory results:

The neutral solution of manganese salt, containing 10 grms. of zinc sulphate, is diluted to 500 c.c. and heated to boiling. One grm. of freshly ignited zinc oxide is then added, and the solution titrated with permanganate, with frequent heating to boiling, and shaking, until the permanganate is no longer decolourised. The solution is then cooled under the tap for a minute or two, 1 to 2 c.c. of glacial acetic acid are added, and the liquid thoroughly shaken. The hot but not boiling liquid is then titrated further with permanganate, added a few drops at a time, with vigorous shaking for half a minute after each addition, until the supernatant liquid retains its pink colour after being well shaken several times. The end-point is then reached, and the pink coloration will persist, as a rule, for one or two hours.

In this form, Fischer's process leaves little to be desired as far as accuracy is concerned, while the remarkable manner in which the precipitate subsides after the addition of acetic acid enables the titrations to be finished with the greatest ease.

The results obtained are summarised in Tables I. to IV. Those given in Tables I., II., and IV., represent mean values, each experiment having been repeated several times, with concordant results. The weights of zinc sulphate specified refer to *anhydrous* salt; it is not clear from Fischer's paper whether he means anhydrous salt or heptahydrate. The results have not been corrected by "blank experiments," which fact may partly account for the rather high results obtained in the presence of large amounts of sodium chloride and zinc sulphate.

Table I. shows that accurate results are obtained when chloride is present to the extent of 10 grms. of sodium chloride; further, that there is little necessity to increase the quantity of zinc sulphate beyond 5 grms. The comparative results of Table II. show that an increase in the amount of chloride present from 10 to 40 grms. of sodium chloride has no influence on the accuracy of the results. Also, that in the total absence of chloride the quantity of permanganate required for titration increases slightly with an increase in the quantity of zinc sulphate present, the results being rather low when little zinc sulphate is added. The quantity of this salt recommended by Fischer (*loc. cit.*, p. 53) seems, however, unnecessarily large, and it is preferable to operate in chloride solution rather than increase the amount of zinc sulphate present beyond 10 grms.

TABLE I.

These values were obtained from manganous chloride, prepared, as described on p. 53, from 50 c.c. of the same $\frac{N}{10}$ permanganate used for the titration.

$KMnO_4$ (theory for titration) = 33·33 c.cm.

$ZnSO_4$. Grms.	NaCl. Grms.	$KMnO_4$. C.C.	$ZnSO_4$. Grms.	NaCl. Grms.	$KMnO_4$. C.C.
5	0	33·40	5	5	33·30
10	0	33·40	10	5	33·45
20	0	33·50	20	5	33·40
5	2	33·30	5	10	33·35
10	2	33·40	10	10	33·45
20	2	33·40	20	10	33·45

TABLE II.

$MnSO_4$ soln. = 6 grms. per litre.
25 c.c. used in each experiment.

$ZnSO_4$. Grms.	NaCl. Grms.	$KMnO_4$. C.C.	$ZnSO_4$. Grms.	NaCl. Grms.	$KMnO_4$. C.C.
10	5	30·55	5	40	30·65
5	10	30·55	10	40	30·60
10	10	30·60	5	0	30·40
5	20	30·55	10	0	30·50
10	20	30·50	15	0	30·50
5	30	30·55	20	0	30·55
10	30	30·60	30	0	30·70

Table III. shows that, by increasing the concentration of the permanganate employed, it is possible to titrate accurately twice the quantity of manganese that can usually be conveniently taken.

TABLE III.

$\frac{N}{5}$ $KMnO_4$ used. Amount reduced = 50·11 c.c.
Theory for titration = 33·41 c.c.

$ZnSO_4$. Grms.	NaCl. Grms.	$KMnO_4$. C.C.
10	10	33·45
10	20	33·50
10	30	33·45
5	0	33·30
10	0	33·45
20	0	33·40
30	0	33·45

TABLE IV.

50·11 c.c. $KMnO_4$ soln. reduced and titrated.
25 c.c. $KMnO_4$ (approx. $\frac{N}{10}$) = 24·66 c.c. $Na_2S_2O_3$.
Theory for $KMnO_4$ = 33·41 c.c.

1. $ZnSO_4$. Grms.	2. NaCl. Grms.	3. $KMnO_4$, C.C. to End-pt.	4. $KMnO_4$, Total Vol. added.	5. $Na_2S_2O_3$, C.C. for Ppt.	6. $KMnO_4$, Equiv. to 5.	7. $Na_2S_2O_3$, C.C. for Filtrate.	8. $KMnO_4$, Equiv. to 4–7.
10	0	33·40	34·50	33·10	33·55	1·05	33·44
10	5	33·45	34·50	32·96	33·41	1·00	33·49
10	10	33·48	34·50	33·05	33·50	0·98	33·51
10	20	33·45	34·50	33·04	33·49	1·00	33·49
10	30	33·55	34·50	33·05	33·50	0·85	33·64
10	40	33·55	34·50	33·05	33·50	0·80	33·69
10	50	33·55	34·50	33·02	33·47	0·89	33·60

Table IV. summarises the results of experiments made to determine the oxidising power of the precipitate. After titrating in the manner previously described (p. 54), a slight excess of permanganate was added to the solution, and the whole shaken vigorously for several minutes. The solution was then cooled, the precipitate filtered off on asbestos, and its available oxygen determined by dissolving in dilute hydrochloric acid and potassium iodide (Pickering, *J. Chem. Soc.*, 1880, **37**, 128), and titrating the liberated iodine with sodium thiosulphate, itself standardised against the permanganate solution. The excess of permanganate in the filtrate was also determined iodimetrically. The results obtained by these two processes are seen to be in substantial agreement with those obtained by direct titration with permanganate.

In applying Fischer's process in actual practice, a difficulty arises unless the volume of permanganate required is known to within 3 or 4 c.c., since, owing to the slowness with which the finely divided precipitate settles in the presence of zinc oxide, the time required for a titration is unduly prolonged, and the first end-point is difficult to observe. The simplest method of avoiding this is to conduct duplicate titrations, as recommended by Fischer. If the acetic acid is added in one case before the addition of any permanganate, a low result is obtained; but the titration is rapidly effected, and the result serves as a useful guide in the titration proper. An alternative procedure to obtain a preliminary result quickly consists in adding several grams of zinc oxide, omitting the zinc sulphate, and titrating in nearly boiling solution. The experiments recorded in Table IV. naturally suggest a backward titration method; the procedure necessary will be obvious from what has been already said.

2. Comparison of Fischer's Method with Other Methods.

The practical value of Fischer's method has been tested by applying it to the estimation of manganese in three specimens of ferromanganese.

The finely divided samples (1 grm.) were dissolved in 25 c.c. strong hydrochloric acid, the solutions oxidised with 10 c.c. nitric acid, and evaporated to a small bulk; 25 c.c. hydrochloric acid were again added, and the evaporation to a small bulk repeated. The solutions were diluted to 300 c.c. with cold water, and zinc oxide added little by little, with shaking, until the precipitate suddenly coagulated. The liquids were then diluted to 1 litre, filtered through dry papers from ferric hydroxide and zinc oxide, and aliquot parts (100 c.c.) of the filtrates measured out and titrated. The error caused by neglecting the volume of the precipitate is inappreciable. The permanganate solution was conveniently made to contain 3·5 grms. per litre, and was standardised against pure sodium oxalate (Sörensen), which is undoubtedly one of the most accurate methods that can be employed for this purpose.

It should be mentioned, in passing, that the commonly-employed zinc oxide method of separating iron from manganese does not always lead to satisfactory results, although it is not possible to assign the reason.

The method outlined above can be easily carried out in an hour and a half, and, of course, a number of analyses can be finished in proportionately less time.

For purposes of comparison, the samples have also been analysed for manganese by the following three well-known methods:

(*a*) *Basic Acetate Separation and Subsequent Gravimetric Estimation of Manganese as* Mn_3O_4.—The separation of the iron was effected twice with ammonium acetate in the usual manner. The manganese precipitate was crushed to powder after a preliminary ignition, and then ignited in the muffle to constant weight. This method appears to give much better results than the statements of some of its critics would lead one to expect (*cf.* Saniter, *J. Soc. Chem. Ind.*, 1894, **13**, 113).

(*b*) *Bismuthate Process.*—This was carried out according to the procedure of Reddrop and Ramage (*J. Chem. Soc.*, 1895, **67**, 268), except that only half the amount of manganese was employed, and ferrous sulphate was substituted for hydrogen peroxide, as recommended by Brearley and Ibbotson (*Chem. News*, 1901,

84, 247). For accuracy, rapidity, and simplicity, this method leaves nothing to be desired.

(*c*) *Pattinson's Process.*—This was carried out as described by Pattinson (*J. Soc. Chem. Ind.*, 1891, **10**, 337); bromine-water was the oxidising agent employed. It was preferred to standardise the bichromate solution employed upon the precipitate obtained from a known amount of manganese chloride (prepared by reducing a weighed quantity of pure permanganate) rather than with iron wire or ferrous ammonium sulphate; the latter procedure led to slightly higher results. It is necessary to point out, moreover, that commercial ferric chloride contains an appreciable quantity of manganese; consequently, if ferric chloride is added to the solution as Pattinson directs, the amount of manganese contained therein* should be allowed for, or an error of 0·4 to 0·5 per cent. manganese may be thereby introduced in dealing with an 80 per cent. ferromanganese. By standardising the dichromate on pure permanganate to which has been added an amount of ferric chloride equal to that used in an estimation, and arranging that the volume of standard solution required shall be approximately equal to that used in an actual analysis, the error due to this cause may be eliminated.

The results obtained are summarised in Table V. The agreement is very satisfactory; the data given are the mean values derived from several concordant analyses, and not "selected" from a number of results. It will be observed that the results obtained by Pattinson's method, employing ferrous ammonium sulphate as standard, are rather higher than those obtained by the comparative method or by other methods. It has not been found possible to duplicate the results obtained by Mr. J. E. Stead,† and which he forwarded after the results by the first three methods mentioned in Table V. had been obtained. It was at his suggestion that Pattinson's method was then tried, with the foregoing results. For very kindly supplying the samples of ferromanganese for analysis, and forwarding his own analytical results for the manganese, the authors wish to express their cordial thanks to Mr. Stead.

In confirmation of the substantial accuracy of the results set out in Table V., the following analytical results are appended (Table VI.). The results for *iron* are probably rather high, since the alloys were dissolved in hydrochloric acid and the iron determined gravimetrically; but they are sufficiently accurate for the purpose. For assistance in estimating the *carbon*, and for the loan of the necessary apparatus, the authors wish to acknowledge their indebtedness to Mr. A. E. Wraight.

Fischer's method has also been tried on two samples of pyrolusite; the results, with comparative data by two other methods, are set out in Table VII. It will be seen that here, again, the agreement between the volumetric methods is very satisfactory, while the gravimetric results are distinctly higher. This is in accordance with Saniter's remarks upon the gravimetric method as applied to ores (*J. Soc.*

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* This is readily determined by boiling with ammonia and a little hydrogen peroxide, dissolving the washed precipitate in nitric acid and a little sulphurous acid, and estimating the manganese by the bismuthate method.

† The results obtained by Mr. Stead were 82·02 per cent., 80·06 per cent., and 60·50 per cent., respectively.

Chem. Ind., 1894, **13**, 113); in the above analyses the manganese was precipitated with bromine and ammonia directly in the filtrate from the basic ferric acetate. The volumetric methods are therefore to be preferred to the gravimetric process, unless the latter is carried out in a more refined manner, taking the necessary precautions to prevent co-precipitation of other metals, and analysing the final precipitate for impurities; it then becomes too tedious for technical work.

TABLE V.

Method of Analysis.	Sample of Ferromanganese.		
		No. 75.	No. 63.
Volhard-Fischer	80·95	79·15	59·47
Bismuthate	80·92	79·15	59·61
Gravimetric	80·98	79·01	59·77
Pattinson (comparative)	80·93	79·12	59·44
„ (standard ferrous ammonium sulphate)...	81·21	79·41	59·65

TABLE VI.

Ferro.	Iron.	Carbon.	Silicon.	Phosphorus.	Manganese.	
					By Difference.	From Table V.
No 80 ...	11·92	6·75	0·50	0·15	80·68	80·95
„ 75 ...	13·48	6·73	0·56	0·21	79·02	79·11
„ 63 ...	33·52	6·03	0·64	0·05	59·76	59·57

TABLE VII.

Sample of Pyrolusite.	Method of Analysis.			
	Volhard-Fischer.	Bismuthate.	Gravimetric.	
			Authors.	Riley and Harbord.
W.	48·75	48·72	49·48	49·63
E.	50·35	50·50	51·33	51·34

The authors' grateful acknowledgments to Mr. J. E. Stead and Mr. E. A. Wraight have been already expressed; it remains, in conclusion, for them to express their thanks to Miss Helen Cunningham for kindly translating von Longi and Camilla's paper, to Messrs. Riley and Harbord for supplying the pyrolusites, together with their own analytical results for the same, and, finally, to Mr. A. Chaston Chapman for the generous help he has afforded them throughout the course of the work.

NOTE ON THE COMPOSITION OF BRITISH WINES.

By EDWARD RUSSELL, B.Sc., F.I.C., and T. R. HODGSON, M.A., F.I.C.

(*Read at the Meeting, December* 7, 1910.)

The absence of any analytical data bearing upon the composition of British wines, and the infrequent submission of these substances under the Food and Drugs Act, led us to obtain a few specimens for the purpose of comparing the analyses with those from two samples collected under the Act.

The wines were supplied to us under ordinary retail conditions of purchase.

The following table gives the composition of the specimens:

GINGER WINE.

	1.	2.	3.	4.	5.	6.	7.	8.
Specific gravity	1027·74	1062·60	1062·47	1088·60	1139·27	1059·37	1060·60	1062·75
Total solids, grms. per 100 c.c.	12·60	21·19	20·80	28·93	43·23	20·09	20·78	21·14
Ash, grms. per 100 c.c. ...	0·183	0·422	0·128	0·123	0·321	0·317	0·306	0·418
Alkalinity (as K_2O), grms. per 100 c.c.	0·013	0·026	0·038	0·056	0·024	0·023	0·110	0·042
Volatile acidity per cent. as acetic acid	0·13	0·09	0·14	0·07	0·05	0·06	0·10	0·07
Fixed acidity per cent. as lactic acid	0·78	0·47	0·35	0·38	0·64	0·54	0·47	0·41
Reducing sugars, grms. per 100 c.c. as dextrose ...	8·49	16·04	16·68	23·96	29·00	16·74	16·75	17·60
Cane-sugar, grms. per 100 c.c.	1·12	0·43	Nil	Nil	7·31	0·06	1·68	0·68
Salicylic acid, grains per gallon	Nil	Nil	7·0	Nil	Nil	0·35	2·0	Nil
Alcohol by weight ...	11·85%	9·29%	8·98%	10·23%	11·31%	9·93%	9·56%	10·15%
,, by volume ...	14·65%	11·52%	11·11%	12·68%	13·99%	12·31%	11·80%	12·58%
Proof spirit	25·65%	19·84%	19·54%	22·27%	24·52%	21·55%	20·78%	22·07%
Nitrogen, grms. per 100 c.c.	0·011	0·022	0·013	0·013	0·028	0·027	0·032	0·018
Proteins, grms. per 100 c.c.	0·069	0·138	0·082	0·082	0·175	0·169	0·201	0·113
Phosphates	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Ethers, grms. per 100 c.c. as acetic ether	0·070	0·018	0·030	0·019	0·067	0·028	0·046	0·060

The samples varied in colour, from a light yellow to a brown, and were free from sediment except in one case in which yeast cells and a few dead acari were discovered.

It will be necessary to obtain a far larger number of analyses before any standard can be formulated; but it seems obvious that in a fermented liquor such as this which is frequently offered to children, the amount of alcohol should not exceed 25 per cent. of proof spirit, and there should be no trace of salicylic acid or other preservatives.

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NOTE ON GORGONZOLA CHEESE.

By EDWARD HINKS, B.Sc., F.I.C.

(*Read at the Meeting, December* 7, 1910.)

GORGONZOLA cheese is a whole-milk cheese made in various Italian provinces, the best cheeses coming from Gorgonzola and its environs. The old name for it was Stracchino. The milk is curdled with rennet when still warm, and drained in hemp cloths, and the paste is "crumbled" and placed in a mould formed of laths of wood with hemp inside. After a few days the wrappings are removed, and the cheese salted by rolling and pressing in salt, this treatment being continued for a number of days. The formed cheese is put aside to ripen—often in caves—preferably at a temperature of from 12° to 18° C., ripening taking from two to six months. The characteristic green mould of this cheese was at first considered a defect; it is now intentionally developed by spearing the cheese to allow access of air. During the ripening Gorgonzola generally develops a reddish-brown crust, which is said to be sometimes imitated by rubbing in oxide of iron or brick-dust. The best cheese is made in early autumn. It appears to have been first introduced into England about fifty years ago.

Gorgonzola cheese is peculiar in that an addition to it of an artificial mineral coat or rind is practised, so that in commerce there are two classes of cheese—the "natural coated" and the "coated" This practice of adding a mineral coating is an old one, and there is evidence that these two varieties have been imported into England during the last twenty-five years: it is said to be the case that before that date all Gorgonzola cheese was thus coated, but it is impossible to speak with certainty as to the nature and weight of the coating in those days. During the last few years, about half the Gorgonzola imported into this country has been naturally coated and half artificially coated.

There is usually a considerable difference between the prices to the purchaser of the two varieties, and, taking an average, the price has lately been from 7½d. to 8½d. per pound for the coated and 9d. to 11d. for the naturally coated, though it is true that the coated is sometimes sold at the higher price.

The mineral coating now always consists, in my experience, of the natural mineral barytes, mixed generally with tallow, though lard is stated to be sometimes used, and coloured, usually on the outer side only, with oxide of iron. This coating or plaster has amounted, in twenty different purchases, to from 16 to 27 per cent. of the whole weight of the cheese, and was from $\frac{3}{16}$ to $\frac{1}{4}$ inch thick.* The coat is applied to the cheese after the natural rind has formed, and the latter may usually be found to a greater or less extent under the former, though very often the cheese immediately beneath the coat has undergone decomposition, resulting in a glue-like slimy layer, in some cases considerably blackened; it is thus entirely an addition, and not present in place of the natural rind. A point that is of importance, in that it might lead to the actual consumption of the mineral, is that the coat is forced into the holes in the cheese, with the result that plugs of barytes were found running into the cheese up to ⅝ inch beyond the inner surface of the rind.

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* The cheeses were carefully divided so as to get a true and fair proportion of coating to cheese.

There is no difference between the manufacture of the cheeses that are to be sold as naturally coated and those that are to receive a coat; the coat is often applied only a few days before shipment, and is at times applied in England.

The composition of the coat of eight samples of cheese was:

Barytes ...	81·0 to 86·7 per cent.	...	average 83·6 per cent.
Fat (tallow)	12·7 to 19·0 „	...	„ 15·6 „
Moisture ...	0·6 to 1·5 „	...	„ 0·8 „

The barytes was the natural mineral, and contained from 91 to 96 per cent. of barium sulphate, the rest being varying proportions of sulphates of calcium, strontium and sodium, of silica and oxide of iron.

The natural coat or rind amounts to little, from 2 to 4 per cent. of the whole cheese if carefully removed, though more than this would as a rule be cut from the cheese. The following is a typical result of the partial analysis of a rind:

	Per Cent.
Fat	3·0
Ash	22·0
Containing—	
Soluble in water	4·7
Chlorine (as sodium chloride)	2·5
Alkalinity (as sodium carbonate)	1·2
Insoluble in water	17·7
Lime	7·6
Phosphoric acid (P_2O_5)	6·3

Starch is often present in the natural rind, and it would not be surprising to find small quantities of barytes in it, as this mineral is sometimes dusted over the surface of the cheese to dry it if it has "sweated"; the starch may be added for the same purpose.

The following are the results of analyses of the interior portion of five Gorgonzola cheeses:

	Water. Per Cent.	Fat. Per Cent.	Protein (N × 6·37). Per Cent.	Ash. Per Cent.
Coated, A ...	32·1	35·2	28·1	4·6
„ B ...	35·9	34·0	24·7	4·5
„ C ...	30·5	36·3	27·9	4·7
„ D ...	35·1	33·3	24·6	5·1
Natural ...	37·3	31·6	—	—

It is interesting to note that a Commission sat at Milan in 1909 and reported that a mineral coating was considered useful but not necessary for Gorgonzola cheese, especially for lengthy transit, following which report the Italian Minister of Agriculture requested the manufacturers to discontinue the use of barium sulphate. The importation of coated cheese into France is prohibited altogether; the prohibition by the United States has been rescinded, and coated cheese can be imported if labelled as such.

DISCUSSION.

Mr. BODMER said that in cheese coated in the manner described fermentation was liable to take place just inside the coating, producing a disagreeable, slimy, malodorous layer. Moreover, the barytes being very heavy, though it might not add very greatly to the thickness of the rind, caused it to constitute a much larger proportion of the total weight of the cheese than was indicated by its appearance to the eye.

Mr. A. E. PARKES said that he had found the fat of Gorgonzola cheese to differ considerably in composition, as indicated by the proportion of volatile fatty acids, from butter-fat and from the fat of ordinary cheese. He had never found, in the case of Gorgonzola cheese, a higher Reichert-Wollney figure than 25 (generally 23 to 25), the Polenske figure being from 3 to 4, and the Valenta number being low. The Reichert-Wollny figure of the fat of Gorgonzola cheese had been given by Allen ("Organic Analysis," vol. iv., p. 242) as 22, but that was the only reference he had been able to find. Gorgonzola cheese, of course, contained a number of moulds, the chief being *Penicillium glaucum*, which secreted a lipase, and it seemed quite likely that the presence of this mould might affect the composition of the fat. At any rate analysts should be cautious in certifying the presence of foreign fat in such cheese until the matter had been more thoroughly investigated.

Mr. H. D. RICHMOND asked if Mr. Hinks had any information as to whether Gorgonzola cheese was made wholly from cow's milk, or whether sheep's or goat's milk was ever employed. He (Mr. Richmond) had an impression that a proportion of sheep's milk was sometimes used. He was also under the impression that the mould of Gorgonzola cheese had been found to be, not *Penicillium glaucum*, but the modification of *Penicillium* identical with that found in Roquefort cheese.

Mr. E. M. HAWKINS, as an illustration of the deceptive appearance of the rind of coated Gorgonzola cheese, said that, in the case of one sample which he had examined, the inspector who purchased it expressed to him the opinion that it was uncoated, whereas the actual weight of the rind was found to be as much as 21 per cent. of the total weight.

Mr. P. A. E. RICHARDS remarked that, in the case of nearly all the samples of Gorgonzola cheese which he had recently examined under the Act, the rind had been almost entirely removed by the vendors. In several cases the small portions left on the cheese consisted largely of barytes.

Mr. HINKS said that in these cases he had not made an extended investigation into the composition of the fat, but as far as he had gone it seemed to be normal. He gathered from such information as could be obtained in London that the milk used was entirely cow's milk. The mould certainly seemed to be a species of *Penicillium*, but he did not feel able to state definitely whether it was or was not *Penicillium glaucum*.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Comparison of Beef and Yeast Extracts of Known Origin. F. C. Cook. (*U.S. Department of Agriculture, Bureau of Chemistry, Circular* 1910, *No.* 62, 1-7.)—Examination of six samples of known origin shows that yeast extracts contain approximately 1 per cent. of ether-soluble material, whilst beef extracts may contain large amounts (6 to 8 per cent.). Sarcolactic acid was found in the beef extracts, but not in the yeast extracts. When all the results were reduced to a fat-free and water-free basis, the following points of difference between yeast and beef extracts were brought out. The total ash of the yeast extracts was in every case higher (27·3 to 30·4 per cent.) than that of the beef extracts (18·2 to 24·9 per cent.). The nitrogen content of the yeast extracts lay between 7·4 and 7·5 per cent., whilst that of the beef extracts in no case fell below 11·5 per cent. The phosphoric acid, whether total, organic, or that found in the ash, is, on the average, higher in yeast extracts than in beef extracts, but the differences are not great. The phosphoric acid remaining in the ash shows the greatest difference—namely, 3·7 to 3·9 per cent. for yeast, and 2·6 to 3·4 per cent. for beef. It is suggested that the above points of difference may be found useful where it is desired to gain some evidence as to the origin of an extract beyond that afforded by the creatinine test. G. C. J.

Digestibility of Bleached Flour. E. W. Rockwood. (*J. Biol. Chem.*, 1910, 8, 327-340; *Chem. Zentralbl.*, 1910, 2, 1833.)—Artificial digestion experiments on unbleached flour and on portions of the same flour bleached with nitrogen peroxide showed that the bleaching causes no loss of digestibility; in certain cases the bleached flour, and the gluten and starch separated from it, were more readily digested by pepsin and pancreatin than was the unbleached flour. Bread made with bleached flour was found to be as digestible as bread prepared with unbleached flour. W. P. S.

Loss of Soluble and Volatile Fatty Acids during the Ageing of Butter. D. Crispo. (*Bull. Soc. Chim. Belg.*, 1910, 24, 436-438.)—Samples of butter, wrapped in parchment paper and enclosed in tin boxes, showed a decrease in their proportion of volatile acids (Reichert-Meissl values) of 2·3 to 4·3, whilst three samples that had been kept for three years showed decreases of 2·6 to 12·8. In the case of melted and filtered butter-fat kept in flasks closed with an ordinary cork for periods of one to three years, eight samples out of twelve showed a slight gain in the proportion of volatile acids, +0·2 to +1·6, whilst one sample remained the same, and three showed a loss, −0·3 to −1·5. The author therefore concludes that dry butter-fat separated from the other constituents of the butter remains sufficiently constant for further analyses for a period of at least three years, when kept under the conditions described. C. A. M.

Toxic Substances in Vegetable Fats and Butters. J. Hertkorn. (*Chem. Zeit.*, 1910, **34**, 1381-1382.)—A substitute for butter, known as "backa-margarine," has recently produced symptoms of poisoning, and this led to an investigation of the possibility of other instances of the kind taking place. In the case of animal fats that have been properly purified there is no risk, whilst most of the compounds sold for the production of a butter-aroma or flavour in margarine are harmless. Recently, however, amyl esters of fatty acids and preparations consisting of the lower fatty acids of the oleic-acid series or their esters have been sold for this purpose; and since these are prepared by the interaction of potassium cyanide with alkyl halides, ketones, etc., by way of nitriles (cyanides, oxycyanides) as intermediate products, they are usually more or less poisonous even when used in the degree of dilution suitable for perfumery. There is also the risk of traces of the intermediate cyanogen compounds being present as impurities, and it is therefore suggested that the use of these flavouring agents in margarine should be prohibited. Poisonous fatty acids of the acrylic series also occur as glycerides in many crude vegetable oils and fats used in the manufacture of margarine. As a rule they may be eliminated, together with other toxic substances, by heating the fat to a high temperature. This should be followed by a treatment with alkali and acid and washing with water, and physiological tests should always be applied to the vegetable fats intended for butter substitutes. C. A. M.

The Valuation of the Antiseptic Properties of Hops. A. J. Brown and G. B. Ward. (*J. Inst. Brewing*, 1910, **16**, 641-664.)—The authors used in their work an organism closely allied to, if not identical with, Van Laer's *Saccharobacillus Pastorianus*, isolated from a sample of acid beer, and which proved to be capable of rapid multiplication in suitable media, and was very susceptible to the toxic influence of hop extract. Five grms. of the hop it was desired to investigate were extracted for one hour in a steam steriliser with 500 c.c. of distilled water. The medium in which growth of the bacillus is to be observed is prepared by digesting equal weights of malt and cold water for two hours, and then filtering, boiling, again filtering and diluting to a sp. gr. of 1030. It is important that this solution should be quite bright. A number of tubes are filled with 10 c.c. of this solution, plugged with wool and again sterilised in a steamer for ten minutes. Amounts ranging from 0·25 c.c. to 2·5 c.c. of the standard hop extract are pipetted into a series of the tubes containing the cold malt extract, and the tubes are again sterilised for ten minutes. After cooling, each tube is inoculated with a loopful of a freshly-grown culture of the bacillus; they are then incubated at 30° C. for eighteen hours and the growth, if any, noted. The "neutral" value is taken as the least volume of hop extract which just prevents growth of the organism in the malt extract, and when this volume is 0·25 c.c. the toxic or antiseptic value of the hop employed is taken as being 100. The whole experiment is carried out in a manner somewhat analogous to a Lintner diastatic determination. Growth in the tubes can be easily seen by noting the characteristic "silky" sheen of the liquid. Using these standards, the toxic value of a hop can be determined from the formula—$a = \frac{25}{x}$ where a = relative toxic value, and x = c.c. required just to prevent growth—*i.e.*, "neutral" value.

Working in this manner, the following values were obtained:

Hop.	"Neutral" Value.	Relative Toxic Value.
Oregon, 1909; very good quality	0·28	90
Marzells (hops from English Golding sets grown in Bavaria)	0·3	83
Columbians, 1909	0·5	50
Farnham, 1909; very good quality	0·75	33
East Kents, 1909	0·75	33
East Kent Goldings, 1909; fair quality ...	0·9	28
Bavarians, 1909; very poor quality	1·2	21
East Kents, 1909; very medium quality ...	1·3	19
Mid-Kents; fair quality	1·3	19
East Kents, "Old olds"	4	below 6

The above order is much in accordance with that in which ordinary empirical methods would place the hops.

A "hop extract" sold as a partial substitute for copper hops was found to be devoid of antiseptic action on the bacillus employed. A good quality sample of Oregon hops was extracted with petroleum spirit and then with ether, and it was found that on applying the method above described to the soft and hard resin thus obtained, the antiseptic value of the soft resin was about 50 times greater than that of the hard resin, whose activity is almost negligible. The extracted hops left in this experiment, when again extracted with a little water, proved to be devoid of antiseptic value. It was noticed that the organism employed was not killed even by prolonged exposure (sixteen hours) to the hop extract, although its development in malt wort is entirely suppressed by a very small volume. H. F. E. H.

New Reaction of Morphine. G. Denigès. (*Comptes rend.*, 1910, **151**, 1062-1063.)—On treating 10 c.c. of a dilute solution (down to 0·03 grm. per litre) of a morphine salt with 1 c.c. of hydrogen peroxide solution (5 to 10 vols.), followed by 1 c.c. of ammonia solution and a single drop of copper sulphate solution (1 to 4 per cent. according to the amount of morphine), the liquid when shaken gives a rose to intense red coloration. The test may be applied to morphine syrups, and by comparing the intensity of the coloration with that given by standard solutions of morphine hydrochloride in sugar syrup a colorimetric estimation may be made. For the identification of the alkaloid in the form of its hydrochloride the dry salt is mixed with 1 drop of a reagent consisting of 1 c.c. of 3 to 4 per cent. copper sulphate solution, 5 c.c. of ammonia solution, and 5 c.c. of water. In the presence of morphine a pronounced red coloration is immediately produced. No reaction is obtained with codeine, thebaine, papaverine, narceine, or narcotine, but similar colorations are obtained with the direct derivatives of morphine, such as oxymorphine and paramorphine, and with its readily hydrolysable esters such as heroine. The copper appears to act as a catalytic agent in the reaction, but its place cannot be taken by iron or manganese. The reaction may also be employed to localise the alkaloid in plants, the morphine being first converted into its hydrochloride. C. A. M.

Detection and Estimation of Nitrates in Milk by Means of the Diphenylamine-Sulphuric Acid Test. J. Tillmans. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, 20, 676-707.)—The author has investigated the diphenylamine-sulphuric acid test for nitrates, and gives the conditions under which this test is capable of detecting very small quantities of nitrate, more particular attention being paid to the detection of nitrates in milk, as a positive reaction in such case leads to the assumption that the sample is adulterated with water containing nitrates. As is well known, the test depends on the development of a deep blue coloration when a nitrate solution is treated with diphenylamine dissolved in sulphuric acid. The reagent recommended by the author is prepared as follows: 0·085 grm. of diphenylamine is mixed with 190 c.c. of dilute sulphuric acid (1 : 3) in a 500 c.c. flask, and a quantity of concentrated sulphuric acid is added; the mixture becomes heated, and the diphenylamine dissolves. Concentrated sulphuric acid is now added in sufficient quantity to make the volume of the mixture nearly 500 c.c., and, after cooling, the whole is diluted to the mark with the concentrated acid. When this reagent is used in the proportion given subsequently, the mixture of reagent and test solution will contain approximately 60 per cent. of sulphuric acid, in which concentration the blue coloration is relatively stable. In applying the test to milk, the latter is treated with calcium chloride, boiled, and the serum separated. Twenty c.c. of the serum are now shaken with 5 c.c. of lime-water and about 25 c.c. of pure ether, this operation being necessary for the removal of traces of milk-fat and proteins which interfere with the reaction. Two c.c. of the reagent are then mixed in a test-tube with 0·5 c.c. of the purified serum, the mixture is cooled, and placed aside for one hour; the blue coloration produced in the presence of nitrates reaches its full intensity after this lapse of time. Serum obtained from milk free from nitrates yields only a yellowish or pink coloration. The presence of chlorides is essential for the detection of small quantities of nitrate by the test; water containing a relatively large quantity of nitrate, but free from chlorides, does not yield a coloration with the reagent. Nitrites also give a blue coloration, which reaches its full intensity almost immediately, and in this case the presence of chlorides is not necessary. Water containing 0·1 mgrm. of N_2O_5 per litre, and milk serum containing 0·25 mgrm. of N_2O_5 per litre, both yield distinct colorations, and it is possible to detect the presence of 5 per cent. of added water in milk by means of the test, if the water contains not less than 10 mgrms. of N_2O_5 per litre. The test may be rendered quantitative by comparing the coloration obtained with those yielded by quantities of water or milk serum containing known amounts of nitrate. Milk containing an abnormal quantity of dirt may yield a nitrate reaction, but the traces of extraneous matters found in ordinary milk do not interfere with the test.

W. P. S.

Essential Oil of Orange Flowers. G. Laloue. (*Bull. Soc. Chim.*, 1910 [iv.], 7, 1101-1107.)—During the autumn of 1909 the second crop of orange flowers was sufficiently large to be collected for the extraction of the essential oil, and the author was thus enabled to make a comparative study of the oil yielded by the flowers produced in May and in October by the same plants. In each case the flowers were extracted with petroleum spirit, and the residue from the extract separated from the colouring matter and wax by distillation under reduced pressure in a current of steam. The total yield of oil from the earlier flowers was 0·7363 kilo., and from the October flowers 0·6636 kilo. per 1,000 kilos. The oils had the following characteristics :

	May.	October.	Oils freed from Methyl Anthranilate.	
			May.	October.
Sp. gr. at 15° C.	0·8899	0·8887	0·8882	0·8784
Refractive index $[n]_{D\,15^\circ\,C.}$	1·478	1·476	—	—
Optical rotation ($l=100$ mm.)	−0° 48′	−4° 6′	−0° 52′	−4° 6′
Solubility in 80 per cent. alcohol...	over 1 vol.	over 1 vol.	over 1 vol.	over 1 vol.
Acid value	0·7	1·0	—	—
Saponification value ...	70·2	95·8	63·0	86·8
Total esters (as linalyl acetate)	24·6 per cent.	33·4 per cent.	21·3 per cent.	29·5 per cent.
Total alcohols (as linalol)	51·0 „	57·0 „	50·7 „	58·0 „
Free alcohols	—	—	34·0 „	34·9 „
Methyl anthranilate ...	3·53 per cent.	2·74 per cent.	—	—
Geraniol and nerol ...	—	—	5·4 per cent.	6·9 per cent.

The dextro-rotation (about +5°) of ordinary neroli oil is shown to be due to the action of the steam in the distillation under the ordinary pressure. The results given above show that the oil produced in May is richer in terpenic esters and combined alcohols than the October oil, whilst the latter contains more of the primary alcohols, geraniol and nerol, and less linalol than the oil extracted from flowers produced at the normal period. C. A. M.

Essential Oil of Schinus Molle (L.). G. Laloue. (*Bull. Soc. Chim.*, 1910 [iv.], 7, 1107-1109.)—The plant *Schinus molle* (L.), which is popularly known as the American pepper plant, occurs abundantly in different areas in Central and South America, and is cultivated in Senegal, Teneriffe, and Algiers. A sample of the oil distilled from the twigs, flowers, and fruit of a plant grown at Grasse and two Algerian samples gave the following results :

	Algerian Oils.		Oil Distilled at Grasse.
	From Leaves only.	From Twigs, Leaves, and Fruit.	
Sp. gr. at 15° C.	0·8658	0·8634	0·8696
Optical rotation ($l=100$ mm.)	+65° 20′	+50° 51′	+46° 13′
Solubility in 90 per cent. alcohol	3 vols.	5 vols.	10 vols. turbid
Acid value	0·7	—	2·1
Saponification value ...	4·1	5·5	10·3
Esters (as linalyl acetate)...	1·2 per cent.	1·9 per cent.	2·8 per cent.

The terpenes, pinene, and phellandrene, were separated by fractional distillation, and a large fraction (about 20 per cent.) appeared to contain sesquiterpenes. The French oil contained more pinene than the Algerian oils. C. A. M.

Essential Oil of Spanish Marjoram (Thymus Mastichina, L.). B. Dorronsoro. (*Chem. Zeit.*, 1910, **34**, 1299.)—Samples of the pure oils from *Thymus mastichina*, grown in Spain in the four years 1898, 1902, 1908, and 1909, were examined. They were transparent, and of a light yellow to greenish colour, and had an odour recalling that of camphor, with a suggestion of thyme. They gave the following results on analysis: Sp. gr. at 20°/15° C., 0·907 to 0·945; refractive index, $[n]_{D, 23^\circ C.}$ 1·4630 to 1·4654; optical rotation, $[\alpha]_{D, 25^\circ C.} = -1{\cdot}4^\circ$ to $+9^\circ$; solubility in 70 per cent. alcohol, 1 : 5 to 1 : 30; saponification value, 12·7 to 18·5; and acetyl value, 29·2 to 45·6. Examination of the fractions obtained by fractional distillation showed that the oil had the following composition: *d*-pinene, 7·0 to 8·0; cineol or eucalyptol, 64·0 to 72·0; phenols, less than 0·1; ketones (?), less than 0·1; esters (linalyl acetate), 4·44 to 6·47; and free alcohols (linalol), 8·2 to 14·1 per cent.

C. A. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Contribution to Study of Chemical Disinfectants. S. Delépine. (*J. Soc. Chem. Ind.*, 1910, **23**, 1344-1354.)—It is known that organisms subjected for a certain time to the action of certain dilutions of mercuric chloride may resist this disinfectant. If a quantity of hydrogen sulphide be added, the suspended growth of the bacteria is resumed. Experiments in this direction (by the silk-thread method) appeared to show that the albuminate of mercury, which is probably formed by the action of this disinfectant on the test organism (*B. coli* in this case), remains localised and superficial when the bacteria are treated with strengths less than 1 : 100; but that even the small amount of toxic product thus formed can bring about the death of the bacterium in sixty minutes when dilutions between 1 : 4,000 and 1 : 2,000 have been used, and in thirty-five minutes after exposure to 1 : 1,000 and 1 : 500 dilutions. Again, the existence of this "antelethal inhibitory period" is well shown by taking threads loaded with spores of anthrax, and leaving them for one hour and for twenty-four hours in emulsions of certain phenoloids. If after one hour the threads are washed in water, transferred to broth tubes, and incubated, no growth usually occurs even in seven days. If the threads after twenty-four hours' contact with the emulsion be inoculated into guinea-pigs, the animals contract anthrax, and die almost as rapidly as those treated with threads not subjected to the action of disinfectant. Thus it is not easy to prove by simple cultural experiments whether a disinfectant in given doses has really brought about the death of the organism. The three main factors in disinfection may be taken to be: (1) The organism; (2) the disinfectant; (3) the associated matter, which are discussed at length in the original paper.

1. *The Organism.*

It is shown to be highly improbable that all the descendants of a non-sporing bacterium are equally resistant to disinfectants. Thus, morphological differences are well known; and in many cases (*e.g.*, *B. typhosus*) the mass of certain individual organisms may be ten times as great as that of the average individual. These differences must affect the relative resistance of the individual bacteria to chemical agents, for mass is an important factor in chemical actions. Thus, the number of bacteria surviving after the addition of a germicidal agent may indicate either the number with which the germicide has not yet combined, because contact has not been possible or sufficiently long, or the number of bacteria which have been able to resist on account of their greater resistance or greater mass. Again, the differences between the resisting powers of an organism and its spores are enormous.

If the individual organisms of a pure culture may vary in resisting power to chemical and physical agents, it is still more probable that this will be the case with different organisms; and account has therefore to be taken of the action of physical agents which influence the action of chemical agents. "Drying" has a differing effect upon organisms (Buckley, Thesis, 1907). Thus, for instance, when exposed on cotton-wool to air dried over sulphuric acid, the *Sp. choleræ* lived only three hours (died in four hours), the *B. pestis* lived twenty-five days (died in thirty-four days), and the *B. diphtheriæ* eighty-one days (died in one hundred and three days).

The *action of heat* upon organisms varies according to the nature of the bacteria, and it is obvious that the influence of temperature upon the germicidal power of a disinfectant may be considerably modified by the action of temperature alone upon the bacteria exposed to the action of the disinfectant. Differences also exist with regard to the resistance of spores to heat and to chemical disinfectants.

Experiments upon these organisms showed that the *B. pestis* is much less resistant to phenol than *B. typhosus*, which latter is less resistant than *B. coli*.

Young agar or broth cultures of *B. coli* are more resistant than older cultures. Experiments designed to show the effect of age on the cultures as regards their resistance to phenol showed evidence of diminished resistance (in solid and fluid media) after the first few days, after which, in broth, the resistance remains almost constant for over two months, and in agar media the alteration of resistance is still more slow. Further experiments showed that *dry*-thread cultures of *B. coli*, kept in the dark for three months at 14° to 18° C., and tested occasionally during this period, remained almost constant in resisting power for seventeen days, when more than half of the threads had lost some of their resistance. At 18° to 22° C. this loss was more clearly shown, this slight difference in temperature causing a measurably larger loss of resistance of the organism to phenol.

2. *The Disinfectant.*

A large number of different classes of chemical products have been suggested and used for disinfecting purposes, but only a few are practically efficient. The table on p. 62 of commercial disinfectants, quoted by the author and published by Woodhead and Vasey (*The Lancet*, 1909, p. 1457), is of interest.

Properties of Chemical Disinfectants.—These, of course, act as protoplasmic poisons, but in different disinfectants the antiseptic action is obviously due to reactions widely different. The mode of action of emulsions of almost insoluble phenoloids is difficult to understand. If these act only when in solution, the toxicity of the soluble part must be very considerable. It seems remarkable that higher cells should be so little affected by these disinfectants, for bacteria are almost invariably more resistant to poisons than more differentiated cells. The application of ultramicroscopy to the study of such difficulties has yielded but disappointing results.

ANALYSIS OF VARIOUS DISINFECTANTS ON THE MARKET (S. A. VASEY).

	Phenol and Phenoloids.	Resins and Fatty Acids.	Water.	Soda.	Potash.	Gelatin.	Dextrin.	Inert Bodies (Hydrocarbons) by Difference.
Cook's Cofectant Fluid	66·27	24·66	6·40	—	2·67	—	—	—
MacDougall's M.O.H. Disinfectant Fluid...	47·13	33·54	5·60	—	2·10	—	—	11·63
Kerol	40·56	19·14	2·40	1·41	—		—	36·76
Okol (Sanitas) ...	48·50	—	38·50	0·20	—		5·90	6·90
Crude carbolic ...	82·65	—	2·80	—	—		—	14·55
Bactox (Sanitas) ...	39·70	16·70	6·00	—	1·88		—	35·72
Medical Cyllin (Jeyes')	32·08	35·11	6·40	—	1·48		—	24·93
Calvert's No. 5 Carbolic A	93·26	—	6·00				—	0·74
Cyllin (bulk sample)...	40·41	31·23	10·40	2·24			—	15·72
Izal	41·35	—	47·14	0·10	—	11·41	—	Traces.
Lysol	50·96	28·12	18·40	—	2·52	—	—	—
Lawes' Fluid	28·20	25·73	13·60	2·60	—	—	—	29·87
Pearson's Fluid ...	20·70	17·35	8·00	3·87	—	—	—	50·08
Jeyes' Fluid (Chemists)	17·80	20·19	6·40	1·60	—	—	—	54·01
Jeyes' No. 2 (Grocers)	5·13	23·21	22·40	1·38	—	—	—	47·88
Krysyl Fluid	14·16	15·22	12·00	2·20	—	—	—	56·42
Zotul	10·00	35·42	21·60	2·49	—	—	—	30·49

The table on p. 63 shows the results obtained from the action of various disinfectants on the *B. typhosus* by the thread and suspension methods. The germicidal power remains practically the same by each method with the soluble disinfectants, but the action of insoluble disinfectants is largely impeded by the protective action of the thread. The results show the relative power of various types of germicide (see also A. H. Burgess, *The Lancet*, June 23, 1900).

Effects of Dilution.—The degree of dilution of the disinfectant, together with the duration of exposure to its action, is an important factor. By prolonged exposure of an organism to a disinfectant it is possible to obtain a germicidal effect with doses which are quite inefficient in shorter exposures; but there is a limit to this, and after a certain dilution has been reached the duration of exposure is immaterial, no germicidal action being observed. The smallest dilution effective after prolonged exposure is termed the *ultimate lethal dilution*.

RESULTS OF TESTS APPLIED SIMULTANEOUSLY TO SEVEN DISINFECTANTS.

Disinfectants.	THREAD METHOD. With *B. typhosus*, 48 hours' culture, on peptone bouillon agar (+5), at 37° C. Solutions of phenol prepared with distilled water. Two threads, 2 cm. long, in 10 c.c. phenol solution. Temperature, 17° C. for 20 minutes' exposure.		SUSPENSION METHOD. With *B. typhosus*, 24 hours' culture, on peptone bouillon (+5), at 37° C., $\frac{1}{20}$ c.c. culture added to 10 c.c. phenol solution. Temperature, 17° C. for 10 minutes' exposure.	
	Minimal Lethal Dilution observed.	Maximal Non-lethal Dilution observed.	Minimal Lethal Dilution observed.	Maximal Non-lethal Dilution observed.
	Parts per 100,000.	Parts per 100,000.	Parts per 100,000.	Parts per 100,000.
Boric acid	— —	$\frac{1}{20}$ = 5,000	— —	$\frac{1}{20}$ = 5,000
Phenol	$\frac{1}{70}$ = 1,428	$\frac{1}{80}$ = 1,250	$\frac{1}{80}$ = 1,250	$\frac{1}{90}$ = 1,111
Lysol	$\frac{1}{90}$ = 1,111	$\frac{1}{100}$ = 1,000	$\frac{1}{200}$ = 500	$\frac{1}{300}$ = 333
Phenoloid A	$\frac{1}{150}$ = 666	$\frac{1}{200}$ = 500	$\frac{1}{500}$ = 200	$\frac{1}{800}$ = 125
Phenoloid B	$\frac{1}{150}$ = 666	$\frac{1}{200}$ = 500	$\frac{1}{500}$ = 200	$\frac{1}{800}$ = 125
Chlorinated lime (32 per cent. available Cl)	$\frac{1}{1600}$ = 62	$\frac{1}{2000}$ = 50	$\frac{1}{2000}$ = 50	$\frac{1}{3200}$ = 31
Mercuric chloride	$\frac{1}{6400}$* = 15	$\frac{1}{10000}$* = 10	$\frac{1}{3200}$* = 31	$\frac{1}{6400}$* = 15

If dilution is pressed beyond the ultimate lethal dose, a degree is reached when disinfectants actually excite the growth of bacteria. Thus, if a thread soaked in mercuric chloride solution be placed on the surface of a gelatin plate sown with *B. coli*, after two days' incubation a clear zone (without organisms) near the thread will be seen, beyond which the gelatin is crowded with colonies, and between these two zones is a narrow, dense marginal zone containing more crowded colonies. Phenol of the strength of $\frac{1}{8000}$ stimulates the multiplication of *B. coli*.

3. *Associated Material.*

Disinfectants which are used up in combining with the organic matter always associated with the organisms, in practical disinfecting obviously may be rendered largely useless, and the important property of phenol and the phenoloid bodies in this connection is that their action is but little affected by the presence of protein and allied matters. The relative value of disinfectants is therefore determined by their action, not on pathogenic bacteria alone, but on the bacteria plus various associated organic matters. A. R. T.

Estimation of Chlorine in Blood. B. Oppler. (*Zeitsch. physiol. Chem.*, 1910, 70, 198–204.)—A portion of the blood (about 10 c.c.) is introduced into a weighing bottle containing a quantity of ammonium oxalate, the bottle and oxalate having been weighed previously; after again weighing to obtain the weight of the blood, the contents of the bottle are rinsed into a stoppered flask and diluted with from ten to twenty times their volume of water, the total volume of water used being measured. A freshly-prepared 1 per cent. solution of metaphosphoric acid is now added from a burette until the solution is rendered distinctly acid to litmus paper; on shaking the mixture the precipitated proteins become red in colour. A further quantity of metaphosphoric acid is next added, the addition being continued as long as a precipitate forms, and, after the lapse of four hours, the mixture is filtered. The chlorine is then estimated in an aliquot portion of the filtrate by precipitation with silver nitrate and weighing the resulting silver chloride. It is recommended that the precipitate of silver chloride be collected on a filter, then dissolved in ammonia and reprecipitated with nitric acid before being finally collected and weighed. The silver chloride may also be dissolved in potassium cyanide solution, and the silver estimated electrolytically. Attempts were made to combine the estimations of chlorine and dextrose in blood, but metaphosphoric acid cannot be used when the sugar is to be estimated, and clarification of the blood with phosphotungstic acid interferes with the estimation of chlorine.

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* Beyond thorough washing and shaking, and the use of large quantities of nutrient fluid, no attempt was made to neutralise the action of the disinfectants at the end of the exposure.

W. P. S.

Estimation of Sugar in Blood. K. Moeckel and E. Frank. (*Zeitsch. physiol. Chem.*, 1910, **69**, 85-88.)—With slight modifications the process described previously by the authors (ANALYST, 1910, **35**, 260) may be applied directly to the estimation of sugar in blood without first subjecting the blood to clarification. Five c.c. of the blood, to which a small quantity of sodium fluoride has been added, are placed in a 100 c.c. flask, and diluted with 40 c.c. of water. Thirty c.c. of colloidal ferric hydroxide solution, diluted previously with an equal volume of water, are added, the solutions are mixed but not shaken violently, a small quantity of powdered Rochelle salt is added, and, after the whole has been well shaken, a sufficient quantity of the diluted ferric hydroxide solution is added to make the mixture up to a volume of 100 c.c. The contents of the flask are shaken for two minutes, and filtered after the lapse of a further five minutes. Fifty c.c. of the filtrate are used for the titration of the sugar, Bertrand's method being employed. The authors find that it is necessary to cool the boiled mixture of sugar solution and Bertrand's solution before collecting the cuprous oxide on a filter. While the solution is hot, a portion of the cuprous oxide remains in a finely divided condition in the liquid, and often passes through the asbestos filter. Only when the mixture is cooled does the cuprous oxide precipitate completely.

L. Michaelis and P. Rona (*Zeitsch. physiol. Chem.*, 1910, **69**, 498) state that they were the first to use colloidal ferric hydroxide for the clarification of blood.

W. P. S.

Detection of Colchicine in Toxicology. H. Fuhner. (*Archiv. exp. Pathol. Pharm.*, 1910, **63**, 357-373; *Chem. Zentralbl.*, 1910, **2**, 1838.)—Colchicine may be isolated from animals poisoned with this substance by extracting the stomach and intestinal contents and the excreta with ether; the residue obtained on evaporating the ethereal extract is dissolved in water, the fat is removed by treatment with petroleum spirit, and the colchicine is finally extracted from the residue with chloroform. It may be identified by means of the coloration it gives with ferric chloride after hydrolysis with hydrochloric acid (Zeisel's test), or by its toxic action on mice and frogs. A white mouse dies within twenty-four hours after having been given 0·1 mgrm. of colchicine, and the same quantity will kill a frog in from two to four days if the frog be kept at a temperature of 30° to 32° C. At this temperature colchicine is about 500 times more poisonous to frogs than it is at the ordinary temperature. W. P. S.

Detection of Amylolytic Enzymes in Fæces. M. Hirschberg. (*Deut. med. Wochenschr.*, 1910, **36**, 1992-1997; *Chem. Zentralbl.*, 1910, **2**, 1726.)—The functional activity of the pancreas may be tested by determining the amylolytic powers of the enzymes (lipase and trypsin) in the fæces, and the following process is recommended for this purpose. A weighed portion of the fæces is well mixed with a 1 per cent. sodium chloride solution, and the mixture is subjected to centrifugal action. The liquid portion is measured, and quantities of it are introduced into test-tubes containing 5 c.c. of 1 per cent. starch solution, each tube in the series receiving a somewhat larger quantity of the fæcal extract than the preceding one. The tubes are then maintained at a temperature of 38° C. for twenty-four hours, and the contents of all the tubes are then brought to the same volume by the addition of water. Iodine solution is now added to each tube, and note is taken of the tube where all the starch has been changed by the least possible quantity of the extract. The diastatic power of the extract may then be expressed in terms of c.c. of extract per 5 c.c. of starch solution. W. P. S.

Preservation of Fæces. P. E. Howe, T. A. Rutherford, and P. B. Hawk. (*J. Amer. Chem. Soc.*, 1910, **32**, 1683-1686.)—Experiments are described which show that when fæces are preserved in a particular manner, no appreciable change, and in particular no change in moisture or nitrogen content, need be feared, even if it becomes necessary to preserve the material for as long as three weeks. The method of preservation consists in collecting the fæces in "ordinary friction-top paint pails," which have previously been rinsed with a 10 per cent. alcoholic solution of thymol, and then dried. The pails are then kept in a cold store until their contents are required. After as long a period as eight months, the moisture will not be found to have changed as much as 4 per cent., whilst the fæces may in the same time lose one-twentieth, but not more, of their nitrogen. The changes recorded in some cases within twenty days of defecation are so small as to be within the limits of experimental error. G. C. J.

Incineration of Microtome Sections. R. E. Liesegang. (*Biochem. Zeitsch.*, 1910, **28**, 413-417; *Chem. Zentralbl.*, 1910, **2**, 1835.)—Experiments were made with the object of localising various elements in microtome sections by incinerating the latter. Whilst in certain cases incineration rendered the structure of the tissue more visible, it was not possible to localise iron, iodine, or phosphates. W. P. S.

Distribution of Fluorine in the Human Body. E. Zdarek. (*Zeitsch. Physiol. Chem.*, 1910, **69**, 127-137.)—The following quantities of fluorine were found in the various organs, etc., of two healthy men who had been accidentally killed. The figures express grms. of fluorine per kilogrm. of dry substance :

	1.	2.
Heart	0·0046	0·0045
Kidneys	0·0154	0·0134
Brain	0·0023	0·0027
Lungs	0·0022	0·0070
Pancreas	0·0082	0·0235
Liver	0·0068	0·0080
Blood (not dried)	0·0035	—
Femur „	0·0029	0·0035
Humerus „	—	0·0045

The fat obtained from the bones contained about one-half of the amount of fluorine present in the bones. W. P. S.

Histological-Chemical Detection of Peroxydase. R. Fischel. (*Wien. Klin. Wochenschr.*, 1910, **23**, 1557-1558; *Chem. Zentralbl.*, 1910, **2**, 1837.)—When a specimen of pus is allowed to dry at the ordinary temperature on a cover-glass, and is then treated with a 2 per cent. sodium benzidinemonosulphonate solution containing 0·01 c.c. of a 0·3 per cent. hydrogen peroxide solution, a blue coloration is produced, and the leucocytes, in particular, are coloured blue. The same result is observed in the case of the myelocytes of bone-marrow, but lymphocytes do not give a reaction unless the quantity of hydrogen peroxide is increased. Red corpuscles from human blood give the coloration when 0·5 c.c. of the hydrogen peroxide solution is employed. Leucocytes may be distinguished from erythrocytes by the fact that the former no longer give a peroxydase reaction after being heated to a temperature of 100° C., whilst the latter are not affected by the heating. W. P. S.

ORGANIC ANALYSIS.

Direct Estimation of Caoutchouc in Vulcanised Rubber Goods. G. Hübener. (*Chem. Zeit.*, 1910, **34**, 1307-1308, 1315-1316.)—In a previous paper (ANALYST, 1909, **34**, 365) the author described a method for the estimation of caoutchouc in vulcanised goods, depending on the estimation of the bromine-combining value and the sulphur of vulcanisation. In the present article a number of examples are given showing the influence exerted by admixtures of foreign substances on the validity of the method. The addition of rubber substitute has no influence, the substitute does not yield an insoluble bromine derivative under the conditions adopted for bromination, and the whole of its sulphur goes into solution, and is calculated as free sulphur. Guttapercha and balata, likewise, have no influence on the results, and yield no insoluble bromine derivatives. In carrying out the bromination with 0·1 grm. of the finely divided sample, it is best to boil the material with a little water in order to wet it thoroughly before digesting with bromine water. Bromine-tion is to be repeated, if necessary, until the particles of material become quite friable on pressure. In the case of the addition of barium sulphate or lithopone as a loading material, the total sulphur is increased by the amount of that present in the barium sulphate, and a correction must be applied for this. The estimation of the barium sulphate may be effected on the ash of the rubber, or may be made by oxidising a separate portion of the sample, first with nitric acid, and then with bromine, filtering and incinerating the residue. If lead oxide and antimony sulphide be also present, the wet method of oxidation converts these into soluble products, and the barium sulphate alone remains. Likewise in the prescribed method of bromination these and similar bodies are entirely soluble, and have no influence on the results. The same applies to calcium salts, even to calcium sulphate. The author has likewise proved that his method is applicable for the estimation of the caoutchouc in regenerated rubber, and, consequently, when such products are present in the sample, the method shows the total caoutchouc, both regenerated and new. Theoretically, the chief objection to the method is the possible influence of proteins on the results, but it is doubtful whether this objection is a valid one in the case of vulcanised goods. J. F. B.

Analysis of Commercial Calcium Cyanamide. N. Caro (and B. Schück). (*Zeitsch. angew. Chem.*, 1910, **23**, 2407-2411.)—Commercial calcium cyanamide (Kalkstickstoff) contains about 21 per cent. of nitrogen, principally (nearly 20 per cent.) as cyanamide, together with about 0·1 per cent. as dicyandiamide, 0·7 to 0·8 per cent. as urea, and 0·6 to 0·8 per cent. in insoluble combinations. The following is found to be the best method for the separate estimation of the cyanamide, dicyandiamide, and urea. Seven grms. of the sample are shaken with 400 c.c. of water for two to three hours at 20° C., and the mixture is made up to exactly 500 c.c., and filtered. In 250 c.c. of the filtrate the cyanamide is precipitated by addition of an excess of an ammoniacal solution of silver acetate (10 per cent. silver acetate, 4 per cent. ammonia), the mixture is made up to exactly 400 c.c. and filtered, and 300 c.c. of the filtrate is collected for the estimation of dicyandiamide and urea. The precipitate contains an amount of silver which varies with the conditions of the precipitation, but the whole of the cyanamide is thrown down. After washing free from ammonia by means of water, the cyanamide in the precipitate is estimated from the nitrogen content as determined by Kjeldahl's method. In the 300 c.c. of filtrate retained for the estimation of the other constituents, the dicyandiamide is estimated as follows: A 10 per cent. solution of caustic potash is added in excess, and the solution boiled until all ammonia has been expelled. As the amount of dicyandiamide is always small, there will usually be enough silver present to precipitate the whole of it, again as a compound of variable silver content. In case of doubt, a little silver solution may be added, but the solution used in this case must not be an ammoniacal one, since the silver compounds of dicyandiamide are soluble in ammonia, a fact on which the previous separation of the cyanamide depends. The mixture is diluted to 400 c.c. and filtered, and the first 300 c.c. of the filtrate preserved for the estimation of urea. The precipitate, which contains the whole of the dicyandiamide, is washed and dried, and its nitrogen content determined by Kjeldahl's method. From this the percentage of dicyandiamide in the original substance is calculated. In the 300 c.c. of filtrate reserved for estimation of urea, excess of silver is precipitated by means of hydrogen sulphide and filtration, and the excess of hydrogen sulphide expelled by a current of carbon dioxide. The solution is diluted to 400 c.c., and an aliquot portion is evaporated, and the nitrogen in the residue determined by Kjeldahl's method and calculated as urea. If in another aliquot portion the urea be estimated by Liebig's method, using a very dilute solution of mercuric nitrate on account of the small amount of urea to be estimated, an identical result will be obtained. That, at least, is the experience of the authors, and they therefore conclude that, apart from cyanamide and dicyanamide, urea is the only other soluble nitrogenous body in the calcium cyanamide produced by the works whose products they have examined.

G. C. J.

Estimation of Cellulose by Simon and Lohrisch's Method. H. Lohrisch. (*Zeitsch. physiol. Chem.*, 1910, **69**, 143-151.)—In reply to Scheunert and Lotsch's criticism of this method (ANALYST, 1910, **35**, 263), the author agrees with the statement that cellulose is, to some extent, attacked by the reagents employed, and admits that the method is not strictly quantitative. At the same time, he has never met with such large deficiencies as mentioned by Scheunert and Lotsch, and considers that the method is quite suitable for the estimation of cellulose in such substances as fæces, etc., provided that control estimations be made. The cellulose obtained is pure and uncontaminated with other substances, and the time required for the estimation is short. In cases where it is desired to ascertain the proportion of cellulose digested during feeding experiments on animals, the method yields sufficiently accurate results, as the cellulose present in the fodder and in the fæces is estimated under exactly similar conditions.

W. P. S.

Estimation of Cyanogen Compounds in the Gases from Coke Furnaces. E. Lecocq. (*Bull. Soc. Chim. Belg.*, 1910, **24**, 439-445.)—The gases are aspirated through two washing flasks charged with a solution prepared by dissolving 2·5 grms. of pure lead acetate in 200 c.c. of a 10 per cent. solution of pure potassium hydroxide. The aspiration of 50 to 100 litres of the gas is continued for six hours on two consecutive days, when the flasks will contain the hydrocyanic and thiocyanic compounds in solution. The insoluble lead sulphide is rapidly separated and washed with a few c.c. of 10 per cent. potassium hydroxide solution. The filtrate and washings are rendered faintly acid with dilute sulphuric acid (1 : 5), the flask being meanwhile kept cold, and are then made up to 500 c.c., shaken, allowed to stand, and filtered from the precipitated lead sulphate. An aliquot portion (400 c.c.) of the filtrate is used for the estimation of the cyanogen compounds. For this purpose the liquid is treated with 10 c.c. of a 10 per cent. solution of a mixture in equal parts of ferrous and ferric sulphates slightly acidified with sulphuric acid, after which potassium hydroxide solution is added, little by little, until a large proportion of the ferroso-ferric salts are precipitated without the reaction becoming alkaline. Under these conditions Prussian blue is slowly formed, and after the liquid has been left for an hour in a warm place it is acidified with 10 to 20 c.c. of sulphuric acid (1 : 5), then heated for fifteen minutes on the water bath, and allowed to stand for several hours. The liquid is now decanted, and the precipitate of Prussian blue washed on a filter, and dissolved in a little sodium hydroxide solution; the solution is then treated, as in Feld's method (*cf.* Colman, ANALYST, 1910, **35**, 295), with magnesium chloride and mercuric chloride to obtain mercuric cyanide. The mixture is distilled with dilute hydrochloric or sulphuric acid, and the hydrocyanic acid absorbed in a solution of sodium hydroxide, which is subsequently titrated by Denigès' method with $\frac{N}{10}$ silver nitrate solution, with potassium iodide as indicator. The filtrate and washings from the Prussian blue are titrated directly by Volhardt's method with $\frac{N}{10}$ silver nitrate solution to obtain the amount of thiocyanate. Or the latter may be estimated colorimetrically with a solution of a ferric salt, a solution of potassium plumbate (containing a known quantity of thiocyanate), which has been subjected to the same treatment as the liquid under examination, being used as the standard for comparison. Analyses of the gases from eight Belgian coke furnaces showed that they contained on the average 0·093 grm. of cyanogen and 0·189 grm. of thiocyanogen per cubic metre.

C. A. M.

Normal Weight of Dextrose. C. A. Browne. (*J. Ind. and Eng. Chem.*, 1910, **2**, 526-527.)—The normal weight of dextrose, as given by different authorities, varies very considerably. For instance, Landolt gives 32·65 grms. for true c.c. and 32·73 grms. for Mohr c.c., whilst Sidersky gives the figures 32·82 grms. for true c.c. and 32·765 grms. for Mohr c.c. The reason for these differences is due largely to the fact that each worker has taken a different specific rotation of dextrose as a basis of calculation. There is no other sugar which shows such a variation in specific rotation for differences in concentration as dextrose. According to Tollens, this value ranges from 52·52 for a 1 per cent. solution to 54·73 for a 50 per cent. solution, which is about the limit of saturation at 20° C. The author considers that a value should be selected as expressing the normal weight, this being that weight of the pure sugar which, dissolved to 100 true c.c. and polarised at 20° C. in a 200 mm. tube, will give a constant reading of 100 on the saccharimeter scale. In the case of impure products, readings of less than 100 will be obtained, and the latter are corrected by a table giving the true percentage equivalent of each division of the saccharimeter scale. The author has converted the formula of Tollens ($[\alpha]_{D\ 20^\circ\ C.} = 52{\cdot}50 + 0{\cdot}018796p + 0{\cdot}00051683p^2$) from a percentage to a concentration basis, the latter form being more easily applied in the calculation of normal weights. The formula thus recalculated reads: $[\alpha]_{D\ 20^\circ\ C.} = 52{\cdot}50 + 0{\cdot}0227c + 0{\cdot}00022c^2$. According to this formula the specific rotation of a dextrose solution containing 32·5 grms. to 100 c.c. is 53·47. The specific rotation of sucrose being 66·5, and its normal weight (metric c.c. standard) 26·00 grms., 32·336 grms. of pure dextrose dissolved to 100 true c.c. will give a reading of 100 on the Ventzke scale. This value is still not absolutely correct, as there is a slight difference in the rotation dispersion of sucrose and dextrose as compared with quartz. The value of 1° Ventzke in angular degrees for dextrose is 0·3448, and the value of the 100 point of the Ventzke scale for dextrose may be taken, therefore, as 34·48 angular degrees. The specific rotation of a dextrose solution of 32·34 grms. concentration, according to the preceding formula, is 53·46. Substituting these values in the general concentration formula for specific rotation, $[\alpha]_{D\,20^\circ\,C.} = \frac{\alpha \cdot 100}{l.c.}$, in which a is the reading in angular degrees, l the dcm. length of the tube, and c the grms. of sugar dissolved to 100 c.c., we obtain for a 200 mm. tube the value: $c = \frac{34{\cdot}48 \times 100}{2 \times 53{\cdot}46} = 32{\cdot}248$ grms., as the correct normal weight of dextrose.

The correction to be added to any reading (s) of the saccharimeter scale is very approximately $+0{\cdot}02s - 0{\cdot}002s^2$. The percentage of dextrose (D) corresponding with any scale reading (s) is therefore expressed by the formula:

$$D = s + 0{\cdot}02s - 0{\cdot}0002s^2.$$

W. P. S.

Methods of Testing the (General) Stability of Explosives in Various Countries. (*Zeitsch. Schiess. u. Sprengstoffw.*, 1910, **5**, 10, 21, 72, 210, 432.)—This interim report of the Commission appointed by the Sixth Congress of Applied Chemistry to inquire into the methods of testing used in various countries, with a view to their unification, deals with the chemical stability, resistance to accidental explosion by shock, and other properties necessary for the safe manufacture, storage, transport, and use of blasting agents, propellants, and other explosives. The tests employed by the British, French, Dutch, Spanish, Italian, United States, and Belgian Governments are described. O. E. M.

Estimation of Phenol and *p*-Cresol in the Presence of Each Other. M. Siegfried and R. Zimmermann. (*Biochem. Zeitsch.*, 1910, **29**, 368-388.)—The method proposed depends on the estimation of the quantity of bromine required, under definite conditions, to convert the phenol into tribromophenol and the *p*-cresol into tribromocresol. It also depends on the estimation of the amount of bromine which is necessary, in the presence of potassium iodide, for the conversion of the two substances into tribromophenol and dibromo-*p*-cresol respectively. From the results of these estimations the quantities of phenol and *p*-cresol present may be calculated. The reagent solutions required are: (1) $\frac{N}{10}$ sodium thiosulphate solution; (2) potassium bromate-bromide solution, containing 0·834 grm. of potassium bromate and 2·97 grms. of potassium bromide per litre (the bromine titre of this solution is determined by titration); (3) a 5 per cent. potassium iodide solution; (4) dilute sulphuric acid (1 : 1); (5) 25 per cent. hydrochloric acid. One hundred c.c. of an aqueous solution containing the two phenols are placed in a 500-c.c. stoppered flask (if 100 c.c. of the solution are not available, water must be added to make up the volume), 30 c.c. of the dilute sulphuric acid are added, and the potassium bromate-bromide solution is run in from a burette until the precipitate which forms coagulates on shaking the mixture, and the solution is coloured distinctly yellow. A further quantity of the bromate solution is then added, so that at least 8 atoms of bromine are present for each molecule of phenol, and the whole is shaken frequently for one hour. After the lapse of this time the solution is filtered through glass-wool, and the filtrate is allowed to run into 30 c.c. of potassium iodide solution; water is added to the flask, shaken to dissolve bromine vapours, and then poured through the filter. The iodine liberated from the potassium iodide is then titrated. This estimation gives the quantity of bromine required to convert the phenol and *p*-cresol into their tribromo compounds. The quantity of bromine may be termed B_1. A second portion of 100 c.c. of the phenolic solution is now placed in a litre flask, 30 c.c. of hydrochloric acid are added, and the mixture is diluted with water to a volume of about 500 c.c. After the addition of the same volume of bromate solution as was employed in the first estimation, the flask is closed and the contents are mixed, but not shaken. At the end of fifteen minutes 30 c.c. of potassium iodide solution are added, the mixture is well shaken, and set aside in a dark place for one hour. The free iodine is then titrated. The amount of bromine necessary for the conversion of the phenols into tribromophenol and dibromo-*p*-cresol is thus obtained. Let it be called B_2, then the amount of *p*-cresol (x) in the solution is found from the equation $x = 0{\cdot}67605\,(B_1 - B_2)$, whilst the amount of phenol (y) is obtained from the equation $y = 0{\cdot}5884.B_2 - 0{\cdot}3923.B_1$. Results of experiments with solutions containing about 0·02 grm. of *p*-cresol and 0·03 grm. of phenol show that the process is trustworthy. W. P. S.

Volumetric Estimation of Quinones by Means of Titanium Trichloride. E. Knecht and E. Hibbert. (*Ber.*, 1910, **43**, 3455-3457.)—Titanium trichloride affords a ready and accurate means for the estimation of quinones, according to the equation—

$$C_6H_4O_2 + 2TiCl_3 + 2HCl = C_6H_6O_2 + 2TiCl_4.$$

The titration may be made either indirectly or directly. In the former case an excess of standardised titanium solution is added to the solution of the quinone in cold water, and the excess of titanium is titrated back by means of a standard solution of iron alum in presence of potassium thiocyanate as indicator. In the second case a trace of methylene blue is added to the quinone solution as indicator, and titration is made directly with the titanium solution. The titanium reacts selectively and quantitatively with the quinone before reducing and discharging the methylene blue. The quantity of the indicator taken should be such that it is decolorised by a single drop of the titanium solution. Results showing 99·7 to 99·8 per cent. of the theoretical values are cited for benzo-, toluo- and *α*-naphtho-quinones; the last, on account of its sparing solubility in water, should first be dissolved in glacial acetic acid and then poured into water. J. F. B.

Estimation of Sulphur in Organic Compounds. T. S. Warunis. (*Chem. Zeit.*, 1910, **34**, 1285-1286.)—The substance (0·2 to 0·4 grm.) is mixed with 10 grms. powdered potassium hydroxide and 5 grms. sodium peroxide in a large crucible of silver or nickel. The crucible, provided with a cover, is placed for about fifteen minutes in an oven maintained at 75° to 85° C., care being taken that the latter temperature is not exceeded at this stage. On removal from the oven the contents of the crucible will be found sintered together, and the crucible is next heated over a small flame, which is gradually increased until complete fusion occurs. It is maintained in this condition for some minutes and then allowed to cool, when the contents are much more easily dissolved by water than is the case when the fusion is made in the ordinary way with soda and peroxide. The aqueous solution of the melt is treated with hydrochloric acid containing bromine, and the sulphur is finally weighed as barium sulphate. G. C. J.

Estimation of Sulphur and Phosphorus in Organic Substances. C. G. L. Wolf and E. Osterberg. (*Biochem. Zeitsch.*, 1910, **29**, 429-438.)—In the method proposed the oxidation is carried out with nitric acid, and subsequently with a mixture of copper nitrate and potassium chlorate. A portion of the substance under examination (urine, etc.) is placed in a flask, and heated with 20 c.c. of fuming nitric acid, until, in the case of solid substances, complete solution is obtained, and nitrous fumes are no longer evolved. Further quantities of nitric acid may be added, if required. The solution is then rinsed into a porcelain basin, 20 c.c. of a solution containing 200 grms. of crystallised copper nitrate and 50 grms. of potassium chlorate per litre of water are added, the mixture is evaporated to dryness, and the residue is ignited at a dull red heat. After cooling, the residue is dissolved in 25 c.c. of boiling dilute hydrochloric acid (1 : 4), the solution is transferred to a flask, 150 c.c. of water are added, the mixture is boiled for fifteen minutes, cooled, and placed aside overnight. The next day the solution is filtered, and the sulphuric acid in the filtrate is precipitated by the addition of barium chloride. The filtrate from the barium sulphate precipitate is then treated with sulphuric acid, the excess of barium is removed by sulphuric acid from the filtrate, and the phosphoric acid is estimated by means of the molybdate method.

The method of oxidising the substance with copper nitrate and potassium chlorate was first recommended by S. R. Benedict (*J. Biol. Chem.*, 1909, **6**, 363), but the authors find that a preliminary treatment with fuming nitric acid is necessary to insure complete oxidation. W. P. S.

Estimation of Total Tartaric Acid by Goldenberg's Method. C. Ordonneau. (*Bull. Soc. Chim.*, 1910 [iv.], **7**, 1034-1041.)—Goldenberg's method (ANALYST, 1908, **33**, 200), which was adopted by one of the sections of the International Congress of Applied Chemistry in 1909, is subjected to criticism as follows: In presence of such proportions of calcium tartrate as occur in crude tartar, the method gives results which are about 2·2 to 2·4 per cent. too low. Salts of aluminium and iron depress the results even more than calcium tartrate, whilst phosphates, except those of aluminium and iron, lead to small positive errors. All these errors can be approximately halved by substituting phenolphthaleïn for litmus as indicator, and it is suggested that phenolphthaleïn should be adopted as indicator in analytical practice connected with the purchase of argol, as it is already used in all works for controlling manufacturing processes. The solution after boiling should be cooled down to 50° C. before commencing the titration, as at this temperature alumina interferes less with the indicator than it does in cold or boiling solution. It is also shown that no purpose is served by adding the acid solution to the potassium tartrate instead of *vice versa*, as recommended by Goldenberg and others. Identical results are obtained when the potassium tartrate solution is added to the acid solution. G. C. J.

INORGANIC ANALYSIS.

Estimation of Acidity in Hydrogen Peroxide Solutions. L. Wöhler and W. Frey. (*Zeitsch. angew. Chem.*, 1910, 23, 2353-2354.)—Endemann has asserted (Analyst, 1909, **34**, 290) that the acidity of hydrogen peroxide solutions is underestimated by almost exactly 50 per cent. when these are directly titrated in the cold with caustic alkali, using phenolphthaleïn as indicator. It is now shown that, with weak solutions of hydrogen peroxide (up to 3 per cent.), direct titration, using phenolphthaleïn as indicator, gives results which are very near the truth (see also Brown, Analyst, 1910, **35**, 497). So far from being low, the results are always a little above the truth, this being due to dissolved carbon dioxide, and, with increasing concentration of hydrogen peroxide, to an acid function of the peroxide. If in titrating weak solutions (up to 3 per cent. hydrogen peroxide) 0·3 c.c. be deducted from the volume of $\frac{N}{10}$ caustic alkali required by 25 c.c., the error of the corrected result will not exceed ±0·1 c.c. $\frac{N}{10}$ acid. Endemann's conclusions were based on a comparison of the direct method with the method in which the peroxide solution is decomposed by prolonged boiling with excess of standard caustic alkali, followed by titration of the excess of caustic alkali, using phenolphthaleïn as indicator. It is shown that this latter method may lead to high results, unless special precautions be taken to protect the boiling solution from the action of carbon dioxide; and it is suggested that Endemann overlooked this fact. In explanation of the alleged anomalous results obtained by direct titration, Endemann (*loc. cit.*) suggested that sodium perhydroxide was neutral to phenolphthaleïn. Were this the fact, it would afford an explanation of unduly high results, but not of the low results of Endemann; but Tafel has shown (*Ber.*, 1894, **27**, 2302) that sodium perhydroxide can be titrated accurately, using phenolphthaleïn as indicator.

If it is required to determine with exactitude the acidity of solutions of hydrogen peroxide of high strength, the hydrogen peroxide must first be destroyed by boiling in platinum, after addition of excess of caustic soda, since the direct method gives rise to errors in excess of 0·4 c.c. $\frac{N}{10}$ solution, when as much as 6 per cent. hydrogen peroxide is present. In such cases methyl orange should be given the preference as indicator, if it is known that no organic and other weak acids are present which would escape measurement by the use of this indicator. If phenolphthaleïn must be used, then the solution must be protected from carbon dioxide during evaporation, or a control experiment must be made. The author does not agree with Schmatolla (*Pharm. Zeit.*, 1905, 641) that Congo red is preferable to methyl orange.

G. C. J.

Separation of Alumina from Ferric Oxide. P. Barbier. (*Bull. Soc. Chim.*, 1910 [iv.], 7, 1027-1028.)—To the solution of alumina and ferric oxide in hydrochloric acid a slight excess of sodium acetate is added, so that the solution assumes a red tint and all the mineral acid present is combined with soda. A 10 per cent. solution of sodium hydrosulphite is added in small portions at a time until the red colour of the ferric compounds disappears. On boiling, the whole of the alumina is precipitated as a dense powder, which is easily filtered and washed free from salts of iron. The filter paper is moistened with nitric acid, ashed, and the alumina added to the contents of the crucible and ignited. The iron is estimated in the filtrate in any convenient manner. No figures are given, but the separation is said to be perfect.

When glucinum is present, this is precipitated as hydroxide with the alumina. The two bases may be separated by prolonged digestion (thirty-six hours) in the cold, with a concentrated solution of ammonium carbonate, which dissolves the glucina. After filtering off the alumina, the glucina in the filtrate may be thrown out of solution by prolonged boiling. C. G. J.

Rapid Determinations and Separations by Means of the Mercury Cathode and Stationary Anode. R. C. Benner and M. L. Hartmann. (*J. Amer. Chem. Soc.*, 1910, **32**, 1628-1636.)—Further experiment has been made with the simple equipment already described by Benner (Analyst, 1910, **35**, 544). Comparison of the rates of deposition under like conditions of current, volume of solution, etc., shows that, although the rate of deposition with the rotating anode is greater than with the stationary anode, it is not sufficiently so to recommend the use of so much more complex a piece of apparatus. The results obtained by Benner's method are as accurate as those obtained by other mercury cathode methods, and with the different metals so far experimented with it is found possible to adopt almost the same conditions as would be chosen if a rotating anode were being used in conjunction with a mercury cathode—that is to say, conditions which determine speedy deposition. Particulars are given concerning more than a hundred experiments, which show that the method is applicable to the estimation of nickel and its separation from aluminium, titanium, and the rare earths, to the estimation of cobalt and zinc, and to the estimation of iron and its separation from aluminium, titanium, and the rare earths. The time required for the deposition of 0·2 to 0·4 grm. from 10 to 20 c.c. of solution varies from ten to twenty minutes, and the results are seldom in error by more than ±0·0005 grm. G. C. J.

Estimation of Chromium in Chrome Tungsten Steel. H. Wdowiszewski and P. Bogoluboff. (*Chem. Zeit.*, 1910, **34**, 1365.)—The method of v. Knorre (*Stahl u. Eisen*, 1907, **27**, 1251) is found to afford the most accurate means of estimating chromium in steels which also contain tungsten, but it is somewhat tedious, and calls for the continuous attention of an experienced operator. The following modification economises time and gives results which differ little from those yielded by the original method of v. Knorre. To 1 to 2 grms. of borings contained in a 500 to 600 Erlenmeyer flask, 10 to 15 c.c. of a 15 per cent. solution of sodium phosphate are added for each grm. of borings, 7 to 8 c.c. of sulphuric acid (sp. gr. 1·65) for each grm. of borings, and finally 5 c.c. water. The borings begin to dissolve at once, and decomposition is complete in fifteen minutes if the contents of the flask be kept moderately warm. The temperature is then raised and 2 c.c. of nitric acid added. The metallic tungsten which separates out at first is oxidised by the nitric acid, and in presence of the sodium phosphate it passes into solution as phosphotungstic acid. The clear solution is diluted with 300 to 500 c.c. of hot water, 3 to 5 grms. ammonium persulphate are added, and the solution boiled until the excess of oxygen is expelled and the whole of the persulphate decomposed. The solution is then cooled, a measured quantity of standard ferrous solution is added, more than is sufficient to reduce the whole of the chromic acid present, and the excess of ferrous salt is determined by titration with permanganate. The whole series of operations occupies less than two hours, and when v. Knorre's method shows 5 per cent. of chromium, the simplified method above described shows 5±0·03 per cent. The quantity of sulphuric acid present should not be less than, nor greatly in excess of, that stated. G. C. J.

Estimation of Hydrogen in Gas Mixtures. O. Brunck. (*Chem. Zeit.*, 1910, **34**, 1313-1314 and 1331-1332.)—The author has used the method of Paal and Hartmann (ANALYST, 1910, **35**, 139), and finds it accurate and convenient. The absorption of the hydrogen may be quickened by using a Hempel pipette similar to that used for phosphorus, but packed with solid glass balls, which are preferable to beads or glass tubing, which are apt to entangle gas bubbles. The regeneration of the palladium sol may be effected as follows: Dilute sulphuric acid is added, until the whole of the palladium, together with its protective colloid (sodium protalbinate), is precipitated. No great excess of acid must be used, or, under the influence of the oxygen of the air, palladium may pass into solution as sulphate. The precipitate, which contains some unchanged picric acid, together with the palladium sol, is washed with water, and then suspended in water, and sodium hydroxide cautiously added until the precipitate disappears and the solution appears homogeneous. Sufficient sodium picrate (the author recommends as much as 5 grms.) is added, and the mixture is again available as an absorbent of hydrogen. G. C. J.

Estimation of Nitrates by the Phenol-Sulphonic Acid Method. H. Caron and D. Raquet. (*Bull. Soc. Chim.*, 1910 [iv.], **7**, 1021-1025.)—Perrier and Farcy have recommended (ANALYST, 1909, **34**, 174) the addition to the standard solution of an amount of chloride equal to that in the water residue under examination. As an alternative means of correcting for the influence of chlorides on the intensity of the coloration produced, they provided a curve having quantities of chlorine as abscissæ, and as ordinates factors by which the apparent amount of nitric anhydride found colorimetrically should be multiplied to get the true amount present. It is now shown that this curve is valueless except for quantities of nitric anhydride closely approximating those employed by Perrier and Farcy in their experiments, since the percentage reduction in colour depends not only on the amount of chloride, but on the absolute amount of nitric anhydride present. A simpler way of overcoming the influence of chlorides is to use freshly-prepared reagent. To 1 c.c. of 90 per cent. phenol, 10 c.c. of sulphuric acid are added, and 1 c.c. of this mixture is used for the test. Chlorine up to 10 parts per 100,000 is then without effect. It is important that the residue from the water and that from the standard solution be treated alike. If the former be cooled in a desiccator, and the latter on the bench, nitric anhydride will be over-estimated; and if the procedure be reversed, it may be under-estimated by more than 50 per cent. G. C. J.

Estimation of Nitrates in Water by a Sulpho-Salicylic Reagent. H. Caron and D. Roquet. (*Bull. Soc. Chim.*, 1910 [iv.], 7, 1025-1027.)—A 1 per cent. solution of salicylic acid in sulphuric acid may with advantage replace the phenol-sulphonic acid of Grandval and Lajoux. The coloration is more intense than that yielded by the ordinary phenol-sulphonic reagent in the proportion of 17 to 10, and is independent of the age of the solution. It is, however, advisable to make use of a comparatively freshly prepared solution for the reasons stated in the preceding abstract. The presence of chlorides does not affect the results, provided the solution be less than five or six days old, whereas phenol-sulphonic acid only one day old gives results which are sensibly influenced by the presence of chlorides. A convenient procedure is to evaporate 10 c.c. of the water under examination with 1 c.c. of 1 per cent. sodium salicylate, to add 1 c.c. of sulphuric acid to the residue, and then, after some minutes, 10 c.c. of water and 10 c.c. of ammonia. G. C. J.

NOTE.—Caustic soda may advantageously replace the ammonia.

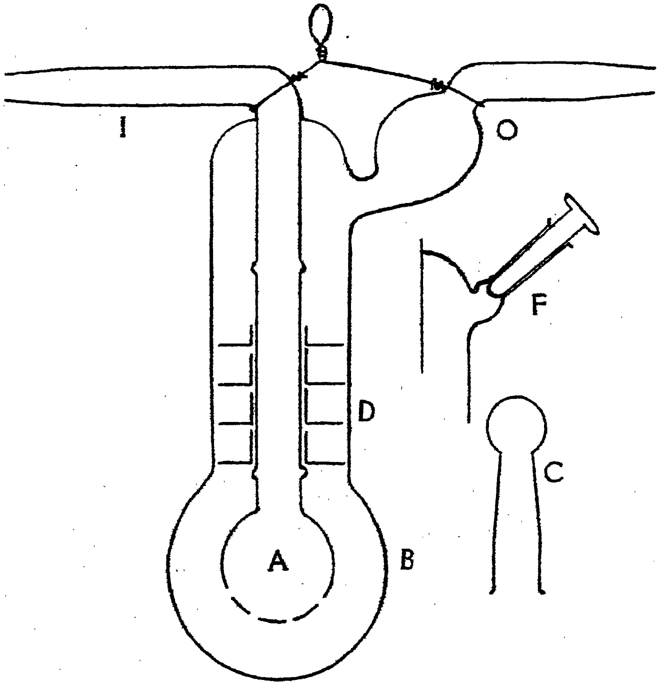
Characteristic Constituent of Natural Vichy Salts. A. Mallat. (*J. Pharm. Chim.*, 1910, **102**, 543-546.)—The mixture of salts obtained by the evaporation of Vichy mineral water, and known by the name of Natural Vichy Salts, always contain lithium salts. The detection of lithia, therefore, in a sample of the salts affords a means of ascertaining whether the salts have been obtained from Vichy water, or whether they consist of an artificially-prepared mixture consisting of sodium bicarbonate and other salts. According to the author's experience, these artificially-prepared mixtures never contain lithium salts. W. P. S.

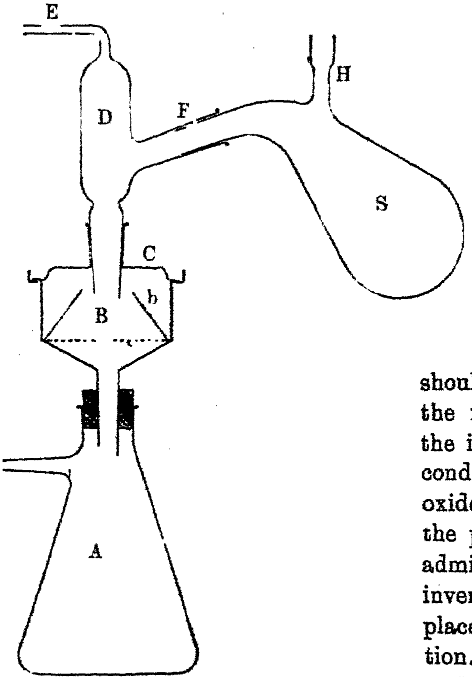
Determination of Hydrated Silicic Acid in Clay. F. K. Pence. (*Trans. Amer. Ceram. Soc.*, 1910, **12**, 43-53; through *J. Soc. Chem. Ind.*, 1910, **29**, 1308.)—Sjollema's diethylamine method (ANALYST, 1903, **28**, 123) and Herman's sodium paratungstate method (*Zeitsch. anal. Chem.*, **46**, 318) of separating amorphous from combined or crystalline silica are said to be too expensive for commercial analysis. The author therefore examined the older methods of separation by caustic soda or sodium carbonate. He finds that caustic soda is useless in this respect, as even a 1 per cent. solution will attack the combined silica. Sodium carbonate, however, gives better results, the method being as follows: Five grms. of the finely ground material are boiled with 120 c.c. of 5 per cent. sodium carbonate solution for ten minutes. After settling for a short time, the solution is decanted, and the sample treated twice more with the carbonate solution. The clay is then transferred to the filter-paper, washed with hot dilute sodium carbonate solution, and the silica in the combined filtrates determined in the usual way by acidification and evaporation to dryness. A control determination should be conducted at the same time, as the vessels usually yield a quantity of silica corresponding to about 0·2 per cent. on a

5 grm. sample. Should the amount of amorphous silica in a clay be unusually large —*i.e.*, more than 3 or 4 per cent.—a smaller sample should be used.

APPARATUS, ETC.

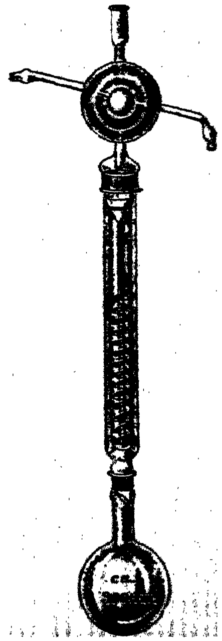
Potash Bulb. C. E. Waters. (*J. Amer. Chem. Soc.*, 1910, **32**, 1691-1693.)—The bulbed tubes A and B are made of very thin "condenser" tubing, the tubes I and O being of ordinary thickness. Around the cylindrical portion of A are four movable discs shown at D. These discs are made by abruptly flaring glass tubing of suitable bore and cutting off about 4 or 5 mm. from the end. Their extreme diameter should be quite 2 mm. less than the internal diameter of the cylindrical part of B, and they should move freely up and down the stem of A. They are prevented from falling below the cylindrical part of B, and from rising within 1 cm. of the outlet, by means of projections blown in the wall of A. The amount of potash solution to be used depends on the rate of flow of the gas, but enough to just reach the second disc is usually sufficient. The gas entering through I is first broken up into small bubbles on passing through the pinholes in the bulbed part of A. It is then trapped in succession by the discs, which, when working properly, alternately rise and fall, continually wetting the walls of B with fresh solution. F illustrates a stoppered side tube for filling the apparatus, to which it is affixed in a plane at right angles to that of the paper. C illustrates the caps which are placed on I and O when weighing. The apparatus is lighter and less fragile than that of Geissler, and has a smaller surface. When filled it weighs 35 to 40 grms. G. C. J.





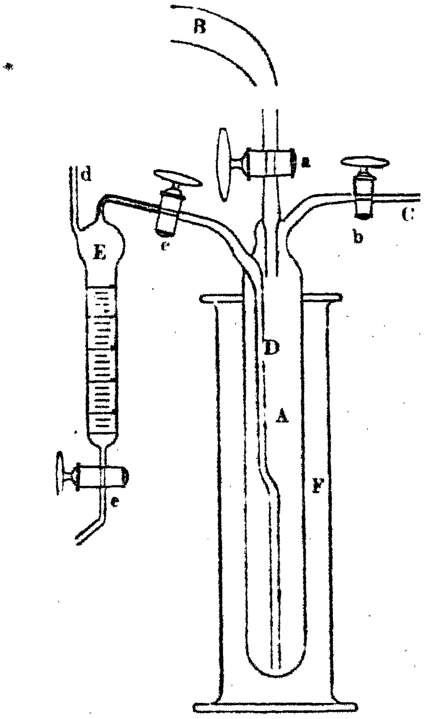
Apparatus for Filtering Hygroscopic Substances or those affected by Air. W. Steinkopf. (*Chem. Zeit.*, 1910, **34**, 1358.)—A suction flask, A, is fitted with a Buchner funnel, B, with an upper vertical extension, upon which rests a ground-glass lid with a central tube. Through this is fitted the ground-in tube of D, which has a side-attachment into which fits the reaction flask G. By turning this flask, the precipitate readily falls into the tube D, and thence on to the filter. The tube H, which should be represented on the nearest side of the flask, and not as shown here, serves for the introduction of a gas or to hold a reflux condenser. Tubes containing phosphorus pentoxide are placed between the suction flask and the pump, and at E, in order to prevent the admission of moisture to the apparatus. An inverted flask, J, with its stem cut off, may be placed upon the filter to keep the paper in position. When it is necessary to exclude all air, an indifferent gas is introduced through E. The apparatus is made by Bender and Hobein, Karlsruhe. C. A. M.

Automatic Extraction of Aqueous Liquids by Means of Specifically Lighter Solvents. R. Kempf. (*Chem. Zeit.*, 1910, **34**, 1365-1366.)—The flask, extraction apparatus, and ball condenser, are connected by means of ground-glass joints. Inside the external tube of the extraction apparatus is a long test-tube provided with glass points near the top and bottom, to insure its taking up an approximately central position within the wider tube. The inner tube, which serves to hold the aqueous liquid to be extracted, is also provided with two holes pierced in its side some way below the top. The only other part of the apparatus is the long funnel tube, around the lower part of which a glass spiral is wound. The operation of the apparatus is as follows: The flask is heated in any convenient manner, and the vapour of the ether or other solvent passes up the annular space surrounding the container of the aqueous liquid, which it maintains at a temperature near the boiling-point of the solvent. On reaching the condenser the vapours are condensed, and the liquid solvent falls into the funnel tube, from the bottom of which it escapes in small globules. These

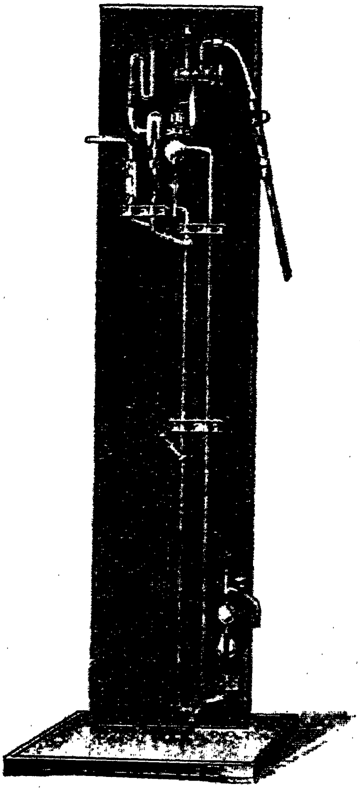


rise slowly through the aqueous liquid, and the glass spiral serves to prolong the period of contact between the immiscible solvents. Finally the ether overflows through the holes near the top of the inner tube, and flows back to the flask. The length of the funnel tube and the effective length of the inner tube (that is, its length from its bottom up to the level of the overflow holes) need to be chosen with due regard to the sp. gr. of the aqueous solution and the other solvent, and to the extent to which water and the other solvent dissolve each other. For ether and aqueous solutions of sp. gr. not far from unity, a funnel length of 52 cm. requires the effective length of the extraction tube to be kept as low as 34 cm. The apparatus is supplied by the makers, the Vereinigten Fabriken für Laboratoriums-bedarf, Berlin, with three inner tubes having effective lengths of 17, 26, and 34 cm., and the longest one serves for the extraction of about 175 c.c. of liquid. G. C. J.

Apparatus for Storing and Measuring Poisonous, Hygroscopic or Volatile Liquids. W. Steinkopf. (*Chem. Zeit.*, 1910, 34, 1319.)—For the storage and measurement of liquids which should not be exposed to the atmosphere, the author has devised the apparatus illustrated in the figure. It somewhat resembles the apparatus described by Falck for the storage of sterilised liquids. It consists of a cylindrical vessel, A, with fairly thick walls, connected with the adapter B by way of the cock *a*; another branch, C, is closed by the cock *b*, whilst a third branch, D, extends nearly to the bottom of the vessel, and is connected by way of the cock *c* with the burette E. This last branch is situated in a plane at right angles to that of the adapter B. The upper portion of the burette is protected by a potash tube which is connected with *d*. The vessel A is suspended in the stout glass cylinder F, in which it is held by a brass lid. The apparatus has been designed chiefly for use with anhydrous hydrocyanic acid. During the process of filling, the tap *c* is closed; *a* and *b* are open, and the open end of C is guarded with calcium chloride and potash tubes. The hydrocyanic acid enters through the adapter B which is connected with the condenser and solidifies in A under the action of an alcohol-carbon dioxide freezing mixture placed in F. After charging, the cocks *a* and *b* are closed, and the acid, if anhydrous, may be kept without change for weeks. For withdrawing a portion of the contents, the tube C is connected with a



calcium chloride tube and a rubber bellows, by which any desired quantity of liquid may be forced over into the burette. The same apparatus may be used with volatile liquids, such as methyl bromide or dry bromine. J. F. B.



Mercury Pump for Rapidly Obtaining a High Vacuum. A. Beutell. (*Chem. Zeit.*, 1910, 34, 1342-1344.)—The apparatus here shown has the advantages of using very little mercury, not being broken by the shock of the falling metal, and of being easily cleaned. The mercury is introduced into the small beaker-like receptacle at the bottom on the right, and this is closed by a stopper provided with a tap. The india-rubber tube at the top is connected with a water-pump, and the mercury is allowed to enter the pump. The tap is then turned to the point fixed by an asbestos stop, so as to allow the necessary amount of air to enter for the mercury to be raised. The mercury will then circulate uninterruptedly for days together through the two vertical columns. To stop the pump, first the lower and then the upper tap is turned 180°, so that air slowly enters, its speed being regulated by the asbestos stops to the taps. Any danger of fracturing the glass by the falling mercury is obviated by allowing a minute quantity of air to enter the pump through the pores of a sound cork fitted in the small tube to the left, so that there is always an air-cushion to protect the glass from the shock. This also prevents the production of electric sparks, and only the ordinary phenomena of the Geissler tube appear. With the most rapid type of the pump, a Röntgen vacuum can be obtained in a connected vessel within fifteen seconds, whilst with the slowest type a minute is required. The amount of mercury needed is from 8 to 20 c.c. The pump may be obtained from F. Hugershoff, Leipzig. C. A. M.

Thermostat for Refractometric Examinations. Poda. (*Chem. Zeit.*, 1910, 34, 1382.)—By means of the apparatus any required temperature may be maintained constant notwithstanding variations in the pressure of the water or gas supply. It consists essentially of a combination of a water-thermostat with a small centrifugal pump. A cylindrical copper vessel holding about 3 litres is nearly filled with water, which is heated by means of a Bunsen burner, controlled by a thermo-regulator. In the water is immersed a Luther's laboratory pump, worked by a water-turbine or an electric motor, so that the water is made to circulate through the prism chamber of the refractometer. The regulator is provided with a [illegible] graduated screw and millimetre scale, so that when once a definite temperature has been established it can rapidly be once more obtained. The copper vessel also contains a cooling coil, so that temperatures lower than that of the room may be obtained. The apparatus is made by K. Grass, Innsbruck. C. A. M.

Modified Separatory Funnel and Washer for Heavy Liquids. H. M. Atkinson. (*Chem. News*, 1910, **102**, 308.)—This is an ordinary pear-shaped separator with tap at the bottom, with the addition of a side-tube carrying a stopcock, about half-way up the side of the separator. It is useful for washing heavy liquids, such as aniline; and the wash-water, caustic soda solution, etc., can be readily run off through the side-tube without running out the main liquid in the funnel. The substance may also be dried with calcium chloride or other reagent without transference.

A. R. T.

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REPORTS.

Report of the Malt Analysis Committee (Coloured Malts and Caramel), 1910, to the Council of the Institute of Brewing. (*J. Inst. Brewing*, 1910, **16**, 529-533.)—The Committee consisted of Messrs. Julian L. Baker, L. Briant, A. J. Brown, A. Chaston Chapman, J. Heron, A. R. Ling, A. Gordon Salamon, and L. T. Thorne, assisted by a number of others connected with the brewing industry.

In the previous Report (*J. Inst. Brewing*, 1906, **12**, 1) it was recommended that the grinding of the malt for analysis should be done in a "Seck" mill set at 25°. For this is now substituted "Seck 0·5 mm.," this being the distance as measured with a feeler gauge between the rollers. The Committee recommend that this mill-setting be taken as applying also in their previous Report. Brown and crystal malts are to be ground in the "Seck" mill, but, owing to the possibility of roasted malts and barleys fouling the mill and colouring the subsequently ground pale malts, it is recommended that black grist be ground finely in a coffee-mill.

Brown and Crystal Malts—*Extract.*—Fifty grms. of the sample are ground in the "Seck" mill as above specified, and the grist mashed with 300 c.c. of distilled water at 158° F. and 100 c.c. of cold water malt extract previously heated to 150° F. This extract is prepared from a well-modified pale malt of diastatic power 30° to 40° (Lintner) by digestion with three times its weight of distilled water for one hour at 60° to 70° F., and subsequent filtration. The mixture is kept for one hour at a temperature of 150° F., then cooled to 60° F. and made up to 515 c.c., shaken, filtered, and the sp. gr. taken. The necessary correction for the cold water malt extract employed is obtained by diluting a volume of 100 c.c. of the malt extract with 300 c.c. of water, the mixture being kept at 150° F. for one hour. Subsequently it is cooled to 60° F., made up to 500 c.c. at 60° F., filtered, and its sp. gr. taken.

Colour.—A volume of 20 c.c. of the above extract, which must be brilliant, is diluted to 100 c.c., and the colour read in a 1-inch cell, using a Lovibond tintometer and glasses of the "52" series.

The colour due to the cold malt extract may be neglected.

BLACK BARLEYS AND MALTS—*Extract.*—A little over 50 grms. of the sample is finely ground in a coffee-mill, and of this exactly 50 grms. are extracted with about 350 c.c. of boiling distilled water, care being taken to avoid balling. The infusion is kept in a bath of boiling water for one hour, after which it is cooled, made up to a volume of 515 c.c. at 60° F., shaken, filtered, and the sp. gr. of the filtrate taken.

Colour.—A volume of 20 c.c. of the above extract, which, if not brilliant, must be refiltered, is diluted to 1,000 c.c., and the colour read in a 1-inch cell, using a Lovibond tintometer and glasses of the "52" series.

CARAMEL—*Colour.*—Ten grms. of the sample are dissolved in distilled water, and made up to 100 c.c. at 60° F. A volume of 10 c.c. of this solution is diluted to 1 litre filtered if necessary, and the colour read in a 1-inch cell, using a Lovibond tintometer and glasses of the "52" series.

STATEMENT OF RESULTS.—Results are expressed to the nearest first decimal place only, except in the case of tint, which is recorded to the nearest half-unit.

Brown and Crystal Malts.—Extract per standard quarter of 336 pounds. Colour: In a 1-inch cell, "52" series Lovibond (2 per cent. extract).

Black Barleys and Malts.—Extract per standard quarter of 336 pounds. Colour: In a 1-inch cell, "52" series Lovibond (0·2 per cent. extract).

Caramels.—Colour: In a 1-inch cell, "52" series Lovibond (0·1 per cent. solution).

H. F. E. H.

Annual Report of the Dominion Laboratory, Wellington, New Zealand (1909). J. S. Maclaurin. (*Wellington, N.Z.*, 1910; pp. 76.)—The report gives, mainly in tabular form, the results of a large number of varied analyses, including analyses of coal, minerals, ores, rocks, gold and silver assays, water, soils, manures, explosives, milk, and other foods and beverages, etc. Amongst the mineral waters examined is one from the lake on White Island, the natural temperature of this water being 110° F. It is remarkable in containing, in addition to free hydrochloric acid (4·83 per cent.) and boron trioxide (0·031 per cent.), pentathionic acid, $H_2S_5O_6$, to the extent of 0·024 per cent.

The milk standard in the Dominion is fixed at 3·25 per cent. of fat and 8·5 per cent. of non-fatty solids, with not less than 12 per cent. of total solids; 1,598 samples were examined in the four worst months of the year to ascertain their quality. The results show that 3·88 per cent. of these samples fell below the standard for milk-fat, thirteen samples containing less than 3·0 per cent.; while 4·13 per cent. were below the standard for non-fatty solids, twenty-one containing less than 8·30 per cent.

A. B. T.

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REVIEW.

TECHNICAL MYCOLOGY. By DR. FRANZ LAFAR. Translated by CHARLES T. C. SALTER. Vol. II., "Eumycetic Fermentation." Part II. With 50 figures in the text. London: Charles Griffin and Co. 1910. Pp. ix, 191-748. Price 18s. net.

This long-expected volume brings to a close the English translation of this valuable work, of which the first volume was published in the original German in 1897. A regrettable delay has occurred in its production, as the corresponding chapters of the original were written in 1904-1906. The treatment of the subject-matter, therefore, is only brought down to a date now five or six years past, and no references later than 1905 seem to be included in the bibliography. How much the book suffers from this unfortunate circumstance is an indication of the rapid progress which is still being made in many branches of the subject. No attempt has been made to remedy this state of affairs, as might have been done by the addition of supplementary paragraphs giving the most recent views on many of the subjects discussed in the text, or even by the insertion of references to them, whereby the value of the book as a work of reference for present use would have been greatly increased.

This said, there is little to add but praise. The book is excellently produced, and editor, translator, and publisher are all to be congratulated on their share in this result. This part includes the bibliography and index to the entire work, together comprising about a quarter of the volume. A special feature of the bibliography, and one greatly to be recommended, is that reference is frequently made, not only to the original paper, but to the abstract in some one or more easily accessible journal, such as the *Chemisches Zentralblatt*, etc. The opportunity of making the bibliography serve also as an index of authors, by adding the number of the page on which each reference is to be found, has been missed, and the work is accordingly without this useful adjunct.

As in previous parts, each section is treated by an acknowledged authority on the subject, with the result that we are presented with a series of thoroughly well-informed monographs.

The range of subject is very wide, and comprises yeast nutrition and culture; the life-history and classification of the saccharomycetes and allied forms; the enzymes of yeast; the chemistry of alcoholic fermentation; and the morphology, classification, and chemical action of the technically important members of the higher groups of fungi, as well as of that lumber room of classification, the *fungi imperfecti*, including the group of Torulaceæ, at the moment of great interest to the English brewer.

One of the most interesting chapters of the book is the account of the life history, variability, and classification of the yeast organisms, in which the fundamental researches of Hansen on these important subjects are described. The present system of classification of the yeasts cannot, however, be regarded as at all satisfactory. This is at once evident from the facts that the Saccharomycetaceæ are defined as spore-producing fungi, the closely allied group of the Torulæ being thus excluded, and that Hansen succeeded by a simple process of cultivation in rendering many species of Saccharomyces permanently non-sporing, without altering any other of their characteristics. Much work is also still called for on the difficult subject of the differentiation of the very numerous species, varieties, and races of yeast.

Analytical methods do not bulk largely in this section of the work, but a considerable amount of information of the greatest possible value to analysts is scattered throughout its pages, particularly in those sections dealing with the mineral foodstuffs of yeast, the production of sulphites by the biochemical reduction of sulphates in fermented liquids, invertase, the by-products of alcoholic fermentation, the fermentation of dextrin by yeast, the autofermentation of yeast, etc.

This volume does not deal in detail with any of the technical processes of the fermentation industries, but it nevertheless provides an almost inexhaustible storehouse of facts bearing directly upon the nature of these processes, the organisms employed in them and the resulting products, and it forms therefore an invaluable weapon in the armoury of those whose fate it is to contend with the difficulties inherent in the employment of living organisms as chemical reagents. A. HARDEN.

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NEW BOOK.

THE CHEMICAL TRADE DIRECTORY OF CHEMICAL MANUFACTURERS, MERCHANTS, BROKERS, AND AGENTS AND MAKERS OF CHEMICAL PLANT IN GREAT BRITAIN AND IRELAND. Fourth Edition. Davis Bros., 265, Strand, London, W.C. 1911. Price 10s.

An 8vo. volume, containing 454 pages of matter. The contents are in three sections. The first, an alphabetical list of names of chemical manufacturers, merchants, brokers and agents, and makers of chemical plant and materials in Great Britain and Ireland, with full addresses. The second, classified lists of chemical products, etc., with names and locations of the makers. The third section, a new one, contains information useful to those in the chemical trade, such as the text of the Alkali Act of 1906, and an abstract of the last report of the Chief Alkali Inspector. Then follow abstracts of legal decisions, the "Census of Production" returns for 1908, and a series of price diagrams, one showing the fluctuations in the price of coal-tar, etc. Tables are given showing exports and imports of chemicals, etc., for the years 1907-1909, the section closing with an abstract of the Factory and Workshop Act of 1901.

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INSTITUTE OF CHEMISTRY.

PASS LIST.

FINAL EXAMINATION FOR THE ASSOCIATESHIP IN THE BRANCH OF BIOLOGICAL CHEMISTRY.—The following candidates for the Associateship passed: F. C. Dyche-Teague, H. F. E. Hulton and H. Shankster. H. Mansfield, A.I.C., presented himself for the Certificate, and passed.

JANUARY EXAMINATIONS, 1911.—Of nine candidates who presented themselves for the Intermediate Examination, six passed: J. Crawford, J. C. Earl, H. J. Evans, W. R. Pratt, D. C. Scott, and G. E. Slim. Of twenty-six candidates who presented themselves for the Final Examination, eighteen passed. In the branch of Mineral Chemistry: W. C. Davis, W. H. H. Norris, B.A. (Cantab.), B.Sc. (Lond.), and R. W. Pope; in the branch of Physical Chemistry: W. R. G. Atkins, B.A. (Dub.), and Bucchok Shen, B.Sc. (Birm.); in the branch of Organic Chemistry: W. Caw, J. C. Duff, B.Sc. (Manc.), A. Greeves, A.R.C.S. (Lond.), B.Sc. (Lond.), W. H. Nodder, B.Sc. (Lond.), T. A. Smith, and T. A. Wallace, B.A. (Dub.), in the branch of the Chemistry of Food and Drugs and of Water: W. H. Dickinson, M.B.Ch.B. (Edin.), Captain I.M.S., H. D. Elkington, R. H. Ellis, A. D. Heywood, and E. H. Shepherd, B.Sc. (Lond.); in the branch of Biological Chemistry: J. Crabtree, B.Sc. (Manc.). Captain Dickinson was examined for the Fellowship. One Fellow, R. W. Clarke, also obtained the Certificate in the Chemistry of Food and Drugs and of Water.

ERRATUM.

Page 1, 1911. Atomic Weight of Hydrogen. *For* 100·8 *read* 1·008.

MARCH, 1911. Vol. XXXVI., No. 420.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The Annual General Meeting of the Society was held on Wednesday evening, February 1, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. W. Voelcker, A.R.S.M., F.I.C., occupied the chair.

The minutes of the previous Annual General Meeting were read and confirmed.

The Hon. Treasurer presented the Accounts of the Society for the year 1910. These were adopted, and votes of thanks were passed to the Hon. Treasurer, Auditors, and Hon. Secretaries.

On the motion of the President, a vote of thanks was passed to the President and Council of the Chemical Society for their kindness in allowing the Society the use of their rooms at Burlington House during the past year.

The President delivered his Annual Address.

On the motion of Mr. Fairley, seconded by Mr. Smetham, a vote of thanks was by acclamation accorded to the President for his Address and for his services in the chair.

Mr. Thomas Macara, F.I.C., and Mr. Cecil Revis, A.C.G.I., were appointed Auditors of the Society's Accounts for the year 1911.

The election of Officers and Council for the ensuing year was postponed until Wednesday, March 1, to which date the meeting was adjourned.

The monthly ordinary meeting followed the annual general meeting, the President, Mr. E. W. Voelcker, occupying the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates in favour of Messrs. R. L. Collett, B.A., A.I.C., W. B. Cowie, F. Evershed, and G. Taylor, were read for the second time; and certificates in favour of Messrs. Joseph Clifford, A.R.C.S.I., assistant chemist in the Survey Department Laboratory, Public Works Ministry Gardens, Cairo; James Connah, B.Sc. (Lond.), F.I.C., Superintending Chemist, Custom House, London, E.C.; Harley F. Knight, "Ledbury," 64, Amhurst Park, Stamford Hill, N., assistant to Mr. Bertram Blount, F.I.C.; Charles H. LaWall, 39, South Tenth Street, Philadelphia, Pa., U.S.A., chemist to the Pennsylvania Food Commissioner (Philadelphia District) and to the Health Commissioner of Philadelphia; William Rest Mummery, F.I.C., 128, Devizes Road, Salisbury, chemist and bacteriologist to the Hygienic Dairy Society, Salisbury;

and W. Macro Seaber, B.Sc. (Lond.), F.I.C., 79, Mark Lane, London, E.C., analytical and consulting chemist, were read for the first time.

Messrs. C. L. L. Claremont, B.Sc., A.I.C., and H. D. Elkington, A.I.C., were elected members of the Society.

The following papers were read: "Note on the Detection and Estimation of Small Quantities of Antimony," by Philip Schidrowitz, Ph.D., and H. A. Goldsbrough; and "Commercial Analysis and Arithmetic," by Clarence A. Seyler, B.Sc., F.I.C.

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ANNIVERSARY DINNER.

THE anniversary dinner of the Society took place on Tuesday evening, January 31, at the Trocadero Restaurant, under the chairmanship of the President, Mr. E. W. Voelcker, A.R.S.M., F.I.C. There were present about one hundred members and guests, among the latter being Sir Thomas Elliott, K.C.B., Secretary of the Board of Agriculture and Fisheries; Mr. W. Whitaker Thompson, LL.B., Chairman of the London County Council; Sir Edward Thorpe, C.B., F.R.S.; Dr. J. J. Dobbie, M.A., F.R.S., Principal Chemist of the Government Laboratory; Professor F. Gowland Hopkins, M.A., D.Sc., F.R.S.; Dr. Frank Clowes, Chemist to the London County Council; Dr. R. T. Glazebrook, M.A., F.R.S., Director of the National Physical Laboratory; Dr. A. T. Morton, Master of the Society of Apothecaries; Mr. Walter F. Reid, President of the Society of Chemical Industry; and Mr. James Swinburne, M.Inst.C.E., F.R.S., President of the Faraday Society.

After the loyal toasts had been duly honoured, the following toasts were proposed: "Government Departments," by Sir Edward Thorpe (responded to by Sir Thomas Elliott and Dr. J. J. Dobbie); "The Society of Public Analysts and Other Analytical Chemists," by Mr. W. Whitaker Thompson (responded to by the President); and "The Guests," by Mr. Otto Hehner (responded to by Mr. Walter F. Reid and Mr. James Swinburne).

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A VOLUMETRIC METHOD FOR THE ESTIMATION OF TUNGSTEN.

BY EDMUND KNECHT AND EVA HIBBERT.

(*Read at the Meeting, December* 7, 1910.)

WHEN tungstic acid or its salts are reduced in strong hydrochloric acid solution by means of granulated zinc, the tungsten passes rapidly to the condition of the blue oxide, then becomes violet, and ultimately yields a brown solution of the tetrachloride. This latter is a powerful reducing agent, and is rapidly and completely brought back again to the condition of tungstic acid by oxidising agents. Otto von der Pfordten (*Ber.*, 1883, **17**, 508) has made use of these reactions for the volumetric estimation of tungsten, employing a standardised solution of potassium permanganate as the oxidising agent, in presence of manganous sulphate. The figures given by this author do not produce the impression that the method is very accurate showing as they do a maximum variation of about 2 per cent.

The new method which we propose is based upon similar principles to that of Otto von der Pfordten, the main differences being that in place of permanganate we use a standard solution of iron alum as oxidising agent (with potassium thiocyanate as indicator), and conduct the titration of the reduced tungsten solution in an atmosphere of carbon dioxide. Furthermore, the titration can be carried out in presence of ferrous iron, which is of course impossible with the permanganate titration. It is consequently not only available for the analysis of commercial tungstic acid (which is extensively used for preparing the metallic filaments in incandescent electric lamps) and tungstates, but also for the rapid and exact estimation of tungsten steels.

If tungstic acid is subjected to energetic reduction by zinc in presence of strong hydrochloric acid, the reaction may be considered as taking place according to the following equation:

$$WoO_3 + 4HCl + H_2 = WoCl_4 + 3H_2O\,;$$

and if the reduced solution be then treated with a ferric salt, reoxidation ensues, according to the equation

$$WoCl_4 + 2FeCl_3 = WoCl_6 + 2FeCl_2.$$

In order to prove this, experiments were first conducted with pure tungstic acid. The purity of the product was ascertained by reducing a known weight in a current of dry hydrogen at a temperature of about 600° C., and weighing the amount of metallic tungsten obtained.

0·5320 grm. of tungstic acid (dried at a red heat) yielded in this way 0·4220 grm. of metallic tungsten. The amount required by theory is 0·4219 grm.

In another experiment, 0·6790 grm. tungstic acid yielded after reduction 0·5385 grm. of metallic tungsten. The amount required by theory was 0·5385 grm. The tungsten obtained in this way when heated in the air to constant weight gave 0·6750 grm. tungstic acid. An accurately weighed quantity of this product was now dissolved in a small volume of ammonia, after which the solution was strongly acidulated with concentrated hydrochloric acid in a flask provided with a Bunsen valve. When reduction was complete, a current of carbon dioxide was passed into the flask, and the solution was titrated with standard iron alum after adding potassium thiocyanate as indicator. The titration gave 99·69 per cent. WoO_3.

The method was further tested on a pure sodium tungstate, with the following results: 0·25 grm. of this product, reduced and titrated in the way indicated, yielded 0·1760 grm. WoO_3. The amount required by theory is 0·1757 grm.

In the reduction of tungstic acid, the contents of the flask almost immediately become intensely blue, and this colour persists for some time. The liquid then assumes, for a very short period, the colour of permanganate, and ultimately yields a brown solution indicating the formation of $WoCl_4$. In titrating with iron alum the brown disappears, and gives place to blue, which persists until the whole of the tungsten is reconverted into tungstic acid. This blue colour may serve as indicator in place of the red of the thiocyanate, identical results being obtained in either case.

Tungsten and Iron.—In order to ascertain whether the estimations of tungsten by the new method were in any way affected by the presence of iron, known amounts of pure tungstate of soda were reduced as above in presence of weighed quantities of iron wire. The results given below are stated in terms of metallic tungsten:

	Taken: Grm.	Found: Grm.
Wo	0·0406	0·0404
Fe	0·9770	—
Wo	0·0203	0·0196-0·0197
Fe	1·0100	—
Wo	0·0944	0·0929
Fe	1·0200	—
Wo	0·1303	0·1292
Fe	1·0020	—

As will be seen, the results are slightly low in all cases.

Ferro-tungsten.—The alloy is ground in an agate mortar and sieved through calico. About 0·1 grm. is weighed in a porcelain crucible, and fused over a Bunsen with about 4 grms. of potassium persulphate until no more particles of metal are visible, which will require about 15 to 20 minutes. The melt is taken up in water, rendered alkaline with caustic soda, filtered from the ferric hydrate, and the tungsten estimated in the filtrate as described above. The iron can be estimated by dissolving the ferric hydrate in hydrochloric acid and titrating with titanous chloride, using potassium thiocyanate or methylene blue as indicator.

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A NEW METHOD FOR THE VOLUMETRIC ESTIMATION OF MOLYBDENUM.

By EDMUND KNECHT and F. W. ATACK.

(*Read at the Meeting, December* 7, 1910.)

The method is based on the reduction of the molybdenum by zinc and strong hydrochloric acid, and the subsequent oxidation of the reduced solution in an atmosphere of carbon dioxide, with standard methylene blue.

Pisani (*Comp. rend.*, 1814, 59, 301) was the first to suggest the method of reducing the molybdenum with zinc and sulphuric acid, and then titrating the reduced solution with permanganate. Many formulæ of a complicated character have been advanced to represent the stage of reduction so obtained—*e.g.*, Mo_5O_7 (von der Pfordten, *Ber.*, 1882, 15, 1929) and $Mo_{12}O_{19}$ (Emmerton, *J. Iron and Steel Inst.*, 1886, 1018). Such formulæ were explained in a paper by Dudley (*Chem. News*, 1898, 77, 195), in which he points to the fact that Mo_2O_3 readily oxidises on exposure to air.

This reduction with zinc and sulphuric acid is very slow and tedious, and, using an atmosphere of carbon dioxide, one fails to obtain results which are in agreement.

Moreover, in all processes involving the use of permanganate, the iron has first to be precipitated if one wishes to estimate the molybdenum directly. Hence a rapid method was sought for the accurate estimation of molybdenum directly in presence of iron.

Energetic reduction with pure granulated zinc and strong hydrochloric acid results in the reduction of molybdenum to the condition of $MoCl_3$ if a current of carbon dioxide be maintained. The stages involved in this reduction are rendered visible by colour changes: the solution first becomes pale green, deepening to blue, then passes to orange, and, after gradually assuming the lighter shade of straw-yellow,* finally turns to a peculiar shade of pink (depending on the amount of molybdenum present), thus indicating the end of the reduction. This final yellowish-pink colour has not been obtained in presence of air, and is best shown if a large excess of strong hydrochloric acid is present.

The reduced solution may now be quantitatively oxidised by methylene blue solution containing 4 grms. per litre, standardised by the method described by Knecht (*J. Soc. Dyers and Col.*, 1905, 21, 9). During this oxidation the salmon-pink passes to a straw-yellow, then to a pale green (in absence of iron), which appears almost, often exactly, halfway through the titration; and, finally, one drop in excess gives the characteristic shade of methylene blue.

In this method of estimation, the oxidation of the $MoCl_3$ does not proceed as far as the stage represented by MoO_3, as is stated to be the case when permanganate is used. It has been shown that the final product is $MoCl_5$, the highest chloride known. Thus, as soon as the oxidation of the $MoCl_3$ is completed as far as $MoCl_4$, the $MoCl_5$ produced gives, in strong hydrochloric acid solution, the pale green colour mentioned above, the colour of the methylene blue appearing when a further volume of methylene blue, equal to that already added, has been run in from the burette. Thus the reaction occurs in two distinct stages—

$$2MoCl_3 + 2HCl + O = 2MoCl_4 + H_2O.$$
$$2MoCl_4 + 2HCl + O = 2MoCl_5 + H_2O.$$

The reduction is effected in an 8-ounce conical flask carrying a three-holed rubber stopper. One of these is fitted with a Bunsen valve, which is not closed as usual with a solid glass bead, but is fitted at the top with a glass tube, into which fits a small piece of indiarubber stopper. Through this passes a platinum wire, to which is attached a rod of pure zinc. Another hole carries a tube for the introduction of a stream of carbon dioxide; the remaining one carries the adapter for the burette. Having placed the molybdenum solution in the flask, 50 to 75 c.c. of strong hydrochloric acid are added, and then a few pieces of pure granulated zinc, the zinc rod being lifted well out of the solution. A violent reduction ensues, during which the stream of carbon dioxide is maintained. After this zinc has completely dissolved, the zinc rod is lowered into the solution for a few moments to insure complete reduction, being then removed, and washed with a little freshly boiled water carefully introduced down the valve from a wash-bottle. The warm solution is now titrated with standard methylene blue.

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* Titration at this stage has shown the straw-yellow solution to approximate to Mo[illegible]O[illegible], which is very near the end of the reduction.

The method was tested on a pure ammonium molybdate, which was found by the gravimetric method (estimation as lead molybdate, $PbMoO_4$) to contain 54·4 per cent. Mo, the amount calculated for the formula $(NH_4)_6Mo_7O_{24}.4H_2O$ being 54·36 per cent. This formula also agrees with the amount of ammonia found—viz., 8·26 per cent. (calc. = 8·28 per cent.); and also with the amount of MoO_3 left on the careful ignition of the finely powdered salt—found, 81·56 per cent. MoO_3 (calc. = 81·53 per cent.).

These results agree exactly with those found, using a recrystallised sample of the ammonium molybdate used above, which had been carefully air-dried—found, 81·70 per cent. MoO_3. By direct titration with methylene blue the following figures were obtained in different estimations: 54·36, 54·43, 54·39, and 54·49 per cent. Mo. The method has also been tested on pure MoO_3 (prepared by the careful ignition of pure ammonium molybdate), which gave 66·60 per cent. Mo, as compared with 66·66 per cent. required by theory. The reduction and titration of molybdenum by this process should not require more than fifteen minutes if ferric iron be absent.

That it is essential to pass a current of carbon dioxide is shown by an experiment in which it was found that the percentage of Mo found by methylene blue was reduced from 54·42 to 44·30 by drawing air through the reduced solution for fifteen minutes. (The colour changes involved in the oxidation were readily followed.)

Molybdenum and Iron.—Ferrous chloride does not reduce methylene blue in hydrochloric acid solution, and hence the method may be used for the direct estimation of molybdenum in presence of iron, provided that all the iron has been reduced to the ferrous condition. The following results were obtained from mixtures of iron and molybdenum:

Molybdenum found: Grm.	Molybdenum taken: Grm.	Iron present: Grm.
0·0347	0·0348	0·0732
0·0368	0·0368	0·10 approx.
0·0367	0·0368	0·15 ,,
0·0368	0·0368	0·15 ,,
0·0449	0·0500	1·0 ,,

In using this method for the analysis of steel, it is to be noted that the salts of the lower oxides of titanium, tungsten, chromium, and vanadium, also exert a reducing action on methylene blue.

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NOTE ON THE DETECTION AND ESTIMATION OF SMALL QUANTITIES OF ANTIMONY.

BY PHILIP SCHIDROWITZ, PH.D., AND H. A. GOLDSBROUGH, A.I.C.

(*Read at the Meeting, February* 1, 1911.)

IN connection with a recent piece of work having an important commercial bearing, it became necessary to work out a process for the determination of small quantities of antimony. By "small" we mean quantities such as $\frac{1}{50}$ or $\frac{1}{100}$ of a grain to the pound. We were not able in the literature on this subject to find any process which appeared likely to assist us under the conditions of our experiments. It did not appear to us that the Marsh process could give practical results, such as are obtained in the case of arsenic, because, as is well known, the deposit occurs on both sides of the flame, thus preventing the formation of a homogeneous mirror. The problem which confronted us was of a twofold nature: (1) The detection and determination of small quantities of antimony in colourless aqueous solutions, and (2) the detection and determination of antimony in certain classes of food-stuffs, such as preserved meats.

Detection and Determination in Clear Aqueous Solutions.—It occurred to us in the first place that it might be possible, in view of the very characteristic colour of the sulphide, to determine roughly the amount of antimony present by comparing the sulphide precipitate with precipitates obtained from solutions containing known quantities. Although relatively very small quantities of antimony may be detected in an aqueous solution in this manner, a few experiments sufficed to show that it was quite impossible to form even a rough opinion regarding quantity by this method. It then occurred to us that, as the antimony sulphide may be readily obtained in colloidal solution, it might be possible to obtain the compound directly in this form by employing a small quantity of a protective colloid. We anticipated that if this could be done we should obtain characteristically coloured solutions in which, by a colorimetric method, the amount of antimony might be readily determined. This we found to be the case. We found that 0·01 grm. of gum acacia in 10 c.c. total volume is sufficient to maintain 2 mgrms. of antimony in colloidal solution. Possibly the amount of antimony that may be held up in this way by this quantity of gum is much larger, but we did not experiment beyond the limit stated. We may add that the antimony employed throughout was in the form of tartar emetic, but that the quantities stated are actual amounts of Sb. The coloration obtained is characteristic and extremely delicate. With regard to the limits, we found that 0·005 mgrm. of Sb in 10 c.c. of liquid gave a distinct coloration—that is to say, it is possible to determine 1 part in 2,000,000. The coloration obtained by passing hydrogen sulphide into hot solutions is deeper than that obtained with cold solutions, and when very small quantities are in question we prefer the hot process. For quantities of 0·02 mgrm., however, the cold process is sufficiently delicate. The control solutions were made up as follows: Known quantities of antimony (in the form of tartar emetic) were made up to a total volume of 10 c.c. with distilled water, after adding

0·5 c.c. of strong HCl and 0·1 c.c. of a 10 per cent. gum acacia solution. It was found that the intensity of coloration was directly proportional to the quantity of antimony present. The experiments on which these statements are based were as follows: (*a*) Solutions containing 0·2 mgrm., 1 mgrm., and 2 mgrms., were directly compared with one another in the colorimeter; (*b*) solutions containing 0·01 mgrm. and 0·02 mgrm. were similarly compared; and (*c*) 0·05 mgrm. and 1 mgrm. were compared. In every case it was found that the heights of the columns of liquid at apparently equal colour intensity were inversely proportional to the amount of antimony present.

The method described presupposes the absence of other heavy metals of the second group which might form colloidal solutions, such as arsenic or mercury. It is also clear that in such cases the method would have to be modified according to circumstances, and that the same applies to cases in which organic matter is present. We have not made any experiments in regard to the separation of antimony from other heavy metals of the second group; but, under the ordinary practical conditions obtaining where food-stuffs are liable to be contaminated by antimony, mercury, lead, arsenic, and copper, are not likely to be present. We may add that arsenic and mercury readily yield colloidal solutions under the same conditions as we have described for antimony.

The Detection and Determination in Other than Clear Aqueous Media.—It is obvious that under certain conditions the method described above cannot be directly applied, and that some method of separating the antimony must be employed. It occurred to us that a convenient method might be that of Reinsch, and then separating the deposited antimony from the copper. It is well known that, in the presence of permanganate, dilute alkaline solutions will dissolve antimony which has been deposited on copper, and that in this way the separation from arsenic or organic matter may be effected. We are not aware, however, that this method has ever been suggested for quantitative work. It seemed to us, however, that under certain conditions the process might give useful results. It was obvious that it would be necessary to employ an excess of permanganate, and that this excess would have to be destroyed prior to passing sulphuretted hydrogen through the final solution. The method which we adopted, and which we found to work fairly well, was as follows: The copper with the Sb deposit was warmed to about 70° C. for a few minutes with 5 c.c. of a 5 per cent. KOH solution, 10 c.c. of distilled water, and an excess of permanganate. As a rule, 1 c.c. of a permanganate solution, of which 1 c.c. was equal to 0·01 grm. Fe, was sufficient. The solution was then poured off the copper, the latter washed with a little distilled water, and the solution then boiled for a few minutes, and subsequently filtered from the precipitated MnO_2. The excess of permanganate in the filtrate was then destroyed by adding a 1 per cent. solution of tartaric acid to the boiling liquid until the latter was colourless. The solution was then made up to a known volume, an appropriate aliquot part taken and made up to 10 c.c., after adding the requisite amount of gum solution and hydrochloric acid, after which the liquid was saturated with H_2S, as described above.

The amounts of antimony recovered in this manner we found to vary roughly between 70 to 80 per cent. of the original amount present. A number of experiments were made by two different observers, the amount actually present being unknown to the individual making the experiment. The following figures are fairly representative of the kind of results obtained:

Observer A. Antimony in Mgrms.		Observer B. Antimony in Mgrms.	
Found.	Present.	Found.	Present.
1·54	2·0	0·20	0·25
0·26	0·35		

These results are, of course, not entirely satisfactory, but they show that, under certain conditions of working, an approximate idea of the amount of antimony present may be obtained. We are doubtful whether any other method existing would give closer results for such small quantities of antimony.

We think the observations we have made might well form the basis of further work on this interesting subject.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Formic Acid in Foods. H. Fincke. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 1-15.)—The author has investigated Auerbach and Pluddemann's method of estimating formic acid by means of mercuric chloride (*Zeitsch. Untersuch. Nahr. Genussm.*, 1909, **17**, 425), and describes processes for the removal of substances which interfere with the accuracy of the method should they occur in the solution in which the formic acid is to be estimated. When a series of estimations has to be made, it will be found most convenient to use the method volumetrically, the formic acid solution being neutralised and heated for two hours in a boiling water-bath with a definite quantity of standard mercuric chloride solution, sodium acetate being also added; after cooling, the excess of mercuric chloride is titrated with potassium iodide solution. The mercuric chloride solution used should contain 58·87 grms. of the salt and 15 grms. of sodium chloride per litre. For occasional estimations the method may be applied gravimetrically, in order to avoid the preparation of standard solutions. In this case the formic acid solution is boiled under a reflux apparatus with sodium acetate and an excess of mercuric chloride, and the precipitated mercurous chloride is collected on a filter and weighed, after having been washed successively with warm water, alcohol, and ether, and dried at a temperature of 100° C.

In separating formic acid from foods, it is necessary to acidify the portion of the sample used with tartaric acid, and to submit it to steam distillation until at least 1,500 c.c. of distillate have been collected. Mineral acids must not be employed, as they may cause any sugars present to caramelise and yield volatile products which reduce mercuric chloride. If formaldehyde or acetaldehyde be present in the food, a flask containing water and calcium carbonate must be placed between the distillation flask and the condenser; the steam and vapours from the distillation are passed through this flask, which is separately heated, and the formic acid is retained by the calcium carbonate, whilst the aldehydes pass over into the distillate. At the end of the distillation the contents of the calcium carbonate flask are filtered, and the formic acid is estimated in the filtrate. When sulphurous acid is present, the distillate (or the filtrate from the calcium carbonate flask, should the food also contain aldehydes) is neutralised, evaporated to a volume of about 100 c.c., and treated with sodium hydroxide and hydrogen peroxide. After standing for four hours at the ordinary temperature, the excess of hydrogen peroxide is destroyed by the addition of freshly precipitated mercuric oxide, and, after the lapse of thirty minutes, the mixture is filtered and the formic acid estimated in the filtrate. Salicylic acid yields an insoluble compound with mercuric chloride in the presence of sodium acetate, but the formation of the compound is prevented by the presence of sodium chloride. Generally, the amount of sodium chloride in the mercuric chloride reagent mentioned above is sufficient for this purpose, but with excessive quantities of salicylic acid it is advisable to concentrate the distillate containing the formic acid, and to add 1 grm. of sodium chloride, 2 grms. of sodium acetate, and 0·5 grm. of mercuric chloride, for every 50 c.c. of the concentrated solution. W. P. S.

Comparison of Meat and Yeast Extracts of Known Origin. F. C. Cook. (*Brewer and Maltster*, 1910, No. 11; through *Wochensch. Brau.*, 1911, **28**, 28.)—The author has submitted beef and yeast extracts to a comparative analytical investigation in order to establish on what points characteristic differences exist. The beef extracts were prepared by grinding lean meat, and extracting with water until the extracts showed no biuret reaction. The united filtrates were evaporated on the water-bath, with periodical removal of coagulated matters. The yeast extracts were prepared by heating well-washed beer yeast, and evaporating in a vacuum pan to a syrupy consistence; such extracts have the appearance and odour of meat extracts, but are somewhat paler in colour. The analytical results are tabulated below. From these it will be observed that, with one exception, the beef extracts are poorer in ash than the yeast extracts. The chlorine is practically constant, and the same is true for the phosphoric acid in the original extracts, but calculated on the dry fat-free solids the yeast extracts are richer in phosphoric acid than the meat extracts. The meat extracts are richer in fat (ethereal extract) and in nitrogen. The chief characteristic difference is the absence of creatine and creatinine from the yeast extracts, and the presence of considerable quantities of these bases in meat extract. This difference affords the best means for distinguishing between the two. Hehner (ANALYST, 1907, **32**, 292) gives the proportion of creatine and creatinine in meat extracts as 10 to 11 per cent.; the author found 8 per cent. as a maximum. No sugar could be detected in any of the extracts. In the ethereal extracts no cholesterol could be detected, and phosphoric acid was absent. Lactic acid could not be detected in the meat extracts, but the acidity of meat extract depends not only on the lactic acid, but also on the phosphates and proteins.

	Yeast Extracts.		Beef Extracts.			
	No. 1.	No. 2.	No. 1.	No. 2.	No. 3.	No. 4.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Dry solids (at 70° C. *in vacuo*)	73·73	72·34	84·78	86·23	79·73	86·66
Ethereal extract	0·83	1·01	6·33	7·85	6·34	6·95
Calculated on dry fat-free solids:						
Ash	30·42	27·32	24·94	20·71	19·93	18·18
Chlorine in the ash (as sodium chloride)	3·27	2·41	2·13	2·64	2·79	2·30
Phosphoric acid:						
In the ash	3·72	3·87	2·93	3·36	2·64	2·79
Total	3·74	3·89	3·19	3·83	2·78	2·85
Organic	0·33	0·41	0·32	0·35	0·27	0·31
Nitrogen:						
Total	7·41	7·45	12·02	12·03	11·76	11·46
Proteoses and peptones	2·95	3·06	4·33	3·88	2·58	3·00
Amino nitrogen	4·46	4·39	7·70	8·15	9·16	8·46
Purine bases	0·80	0·90	—	0·83	1·05	0·80
Creatine	—	—	5·10	3·70	2·06	2·44
Creatinine	—	—	2·83	3·32	3·20	3·28
Acidity (c.c. $\frac{N}{10}$ per grm.)	13·52	14·40	14·31	13·45	13·82	15·47

J. F. B.

Estimation of Citric Acid in Milk. E. Desmoulière. (*Bull. d. Sciences Pharmacol.*, 1910, **17**, 588-594; through *Chem. Zentralb.*, 1910, **2**, 1951-1952.)—The method of Denigès for the detection of citric acid by means of mercuric nitrate and permanganate cannot be made to serve for its quantitative estimation. Methods depending on the extraction of the dry solids of milk with ether after addition of sulphuric acid give low results, unless special precautions are taken, since the ether extracts not only citric and acetic acids, but phosphoric acid, which even at slightly elevated temperatures attacks citric acid. The following method is recommended:

Two hundred c.c. of milk are boiled for a short time under a reflux condenser with 100 c.c. of 2 per cent. acetic acid. When cold, the mixture is filtered, and 150 c.c. of the filtrate are taken down to a pasty consistency on the water-bath after addition of 2 to 3 grms. of kieselguhr. To the cooled residue, not more than 3 c.c. of dilute (1 : 5) sulphuric acid is added, and the mixture allowed to stand for two or three hours, with occasional stirring. A further 3 grms. of kieselguhr are then stirred in, and the mixture repeatedly extracted with cold ether which has been previously saturated with water. The ethereal extracts, measuring about 1,000 c.c. in all, are united, and the ether is distilled off at as low a temperature as possible. The residue is taken up with water, and the solution made up to a known volume. In one portion the total acidity is determined, in another the volatile acids (acetic acid), and in a third the small quantity of phosphoric acid which may possibly be present, even when the directions given are followed closely. The citric acid is found by difference.

In treating human milk, or the milk of goats, sheep, and asses, the amount of dilute sulphuric acid used may with advantage be reduced to 2·5 or even to 2 c.c., but cow's milk may require nearly the full 3 c.c. The following average percentages of citric acid in milk of various origin are given: Cow's milk, 0·21; goat's milk, 0·14; sheep's milk, 0·11; ass's milk, 0·10; and human milk, 0·08. G. C. J.

Estimation of Nicotine in Concentrated Tobacco-Juice. J. Schröder.

(*Chem. Zeit.*, 1911, **35**, 30.)—The concentrated tobacco-juice, now so extensively used for sheep-washes, is usually bought without reference to the proportion of nicotine it contains. Even in cases where a specified proportion of the alkaloid is guaranteed, there is but little agreement between the results obtained by the different published methods, and the author therefore urges that the method used ought always to be stated. Various German wholesale importers were anxious that their samples should be tested by the following method, stated to be due to Ulex: Ten grms. of the tobacco extract are triturated with 25 grms. of calcium oxide and 75 grms. of calcium sulphate, and the mass allowed to stand for three hours over sulphuric acid. The powder is then treated with 3 litres of water containing 25 grms. of sodium hydroxide, 2 litres of the liquid separated, 500 c.c. thereof distilled, and the nicotine in the distillate titrated with $\frac{N}{10}$ hydrochloric acid, with litmus as indicator. Two samples tested by this technical method and by other standard methods gave the following percentages of nicotine:

	Guaranteed Percentage.	Kissling's Method.	Heut's Modification of Kissling's Method.	Keller's Method.	Technical Method.
1 ...	8–9	6·24–6·41	6·45–6·60	6·93–6·69	9·59–9·36
2 ...	12–13	8·64–8·70	8·90–9·20	11·90–12·27	13·45–13·55

Even the method of Toth (ANALYST, 1902, **27**, 12), which Porchet and Régis (ANALYST, 1909, **34**, 534) recommended as sufficiently accurate for technical purposes, may show deviations of as much as 20 per cent. upon the proportion of nicotine present, and in the author's opinion it would be advisable to appoint a committee to work out a reliable method of estimation for international use. C. A. M.

Note.—Evidently the technical method estimates other basic substances besides nicotine.

Estimation of Cocoanut Oil in Lard by Means of the "Ethyl Ester Value." J. Hanuš and J. Thian.

(*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **20**, 745-749.)—Results of an investigation are recorded which show that the "ethyl ester value" (see ANALYST, 1907, **32**, 89; 1908, **33**, 281) of a sample of lard affords reliable evidence as to the presence or absence of cocoanut oil in the sample, and that the value gives approximately the quantity of this oil which is present. W. P. S.

Fatty Oil and Wax obtained from Coffee Berries. H. Meyer and A. Eckert.

(*Monatsh. Chem.*, 1910, **31**, 1227-1251.)—When unroasted coffee berries are extracted with benzene, they yield a mixture consisting of a fatty oil and a wax, and these two constituents may be separated from one another by treatment with petroleum spirit, in which solvent the wax is practically insoluble. The authors find that the oil has a saponification value of from 160 to 182, and an iodine value varying from 90·1 to 91·2. The fatty acids liberated from the oil were found to consist of carnaubic acid, 10 per cent.; daturic acid, 1 to 1·5 per cent.; palmitic acid, 25 to 28 per cent.; capric acid, 0·5 per cent.; oleic acid, 2 per cent.; and linolic acid, 50 per cent. The wax, when freed from fatty oil and caffeine, is a resin-like substance which may be powdered; it is readily soluble in hot alcohol, benzene, chloroform, glacial acetic acid, and acetone, slightly soluble in ether, and almost insoluble in petroleum spirit. It does not crystallize from its solutions. An investigation of the character of the wax showed that it contains carnaubic acid combined with a resin-alcohol (tannol). W. P. S.

Moroccan Olive Oil. A. Sasserath. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **20**, 749-750.)—It is very probable that the oil known as Moroccan olive oil is not a true olive oil, but is obtained from the argan-tree (*Arganum sideroxylon*). This tree somewhat resembles the olive-tree, but the branches are covered with thorns; it yields a fruit similar to olive fruit, from the kernels of which the oil is obtained. A specimen of argan oil, extracted by means of ether from kernels of argan fruit, gave the following results on analysis: Sp. gr. at 15° C., 0·9188; iodine value, 95·94; acid value, 0·18; saponification value, 192·12; Reichert-Meissl value, 1·8; insoluble fatty acids, 95·6 per cent. The oil is golden-yellow in colour, tastes like earthnut oil, and gives a red coloration when shaken with concentrated nitric acid; it does not yield a coloration with either Halphen's or Baudouin's reagents. W. P. S.

Reaction of Turpentine Oil, Wood Turpentine Oil, and Rosin Essence. C. Grimaldi. (*Chem. Zeit.*, 1911, **35**, 52.)—These products may be distinguished from one another by their different behaviour with mercuric acetate solution and by the characteristics of the resulting precipitates. Thus, the precipitate given by turpentine oil is completely soluble in nitric acid, whereas those given by wood turpentine oil or rosin essence ("turpentine essence") are only partially soluble, this difference being probably due to the different terpenes of which the two latter oils almost exclusively consist. The reagent is prepared by mixing 27·5 grms. of mercuric acetate with sufficient water to make up 110 c.c., and filtering the liquid. In applying the test, 1·5 c.c. of the distilled oil are shaken at frequent intervals for some hours in a stoppered 200 c.c. flask with 100 c.c. of the reagent. After the flask has stood for forty-eight hours, 60 c.c. of nitric acid (3 parts by volume of acid of 1·40 sp. gr. to 1 part of water) are introduced, the shaking continued for some time, and the flask again allowed to stand. In the case of pure turpentine oil a clear solution is obtained, whilst wood turpentine oil and rosin essence give turbid liquids from which flocculent precipitates containing mercury slowly deposit. In doubtful cases the test is repeated upon larger quantities. The test may also be applied to mixtures of turpentine oil containing, *e.g.*, 25 per cent. of the other products. Thus, an unmistakable reaction was obtained by treating 8 c.c. of such a mixture with 400 c.c. of the mercuric reagent, and subsequently adding 320 c.c. of the dilute nitric acid (*cf.* ANALYST, 1910, **35**, 412). C. A. M.

Some Indian Oils and Fats. A. Kesava-Menon. (*J. Soc. Chem. Ind.*, 1910, **29**, 1428-1431.)—The author has examined the following authentic samples of Indian oils and fats:

GHI, the melted and clarified butter-fat, freed from water, of the milk of cows, buffaloes, etc. Ghi is much adulterated with the common vegetable oils. The following figures were observed (*cf.* Bolton and Revis, ANALYST, 1910, **35**, 343):

	Cow Ghi (from Milk of Two Cows).	Buffalo Ghi.
Fat:		
Sp. gr. 100°/100° C.	0·8961	0·8965
„ 100°/15° C.	0·8616	0·8619
Acid value	1·49	2·00
Saponification value	218·25	206·80
Reichert-Wollny value	25·70	18·24
Titration number of insoluble volatile acids (c.c. $\frac{N}{10}$ KOH)	2·07	0·74
Unsaponifiable matter, per cent.	0·59	0·73
Butyro-refractometer, at 40° C.	40·6°	44·5°
Fatty Acids (total insoluble):		
Per cent.	88·05	90·65
Titer test	39·75	45·3
Neutralisation value	210·4	204·9
Mean molecular weight	266·6	273·8
Iodine value	32·8	30·7

Note.—The buffalo ghi differs so much from the figures given by previous observers for the fat of buffalo milk that it is probable that, if not an adulterated sample, it is quite abnormal.

BASSIA OILS.—Oils from five trees of this family have been examined, and the following results obtained. The fats from these sources are sold under the names of illupi, ellupai, mowa, mowrah, etc. Arachidic acid is absent. The oil was extracted with petroleum ether, as the ether extract contains much resinous matter.

Name.	Constants of Bassia Oils.							Butyro-Refractometer, Degrees at 45° C.	Unsaponifiable Matter, per Cent.	Iodine Value of Unsaponifiable Matter.
	Sp. Gr. 100°/15° C.	Melting-Point, ° C.	Acid Value.	Saponification Value.	Reichert-Wollny Value.	Titration Number of Insoluble Volatile Acids. c.c.	Iodine Value.			
Bassia butyracea	0·8580	43-51	66·4	194·6	1·25	0·54	41·2	42·5	2·20	77·24
B. latifolia	—	47·5	153·0	187·6-190	1·60	0·36	50·4	48·0	2·27	—
B. longifolia	—	26-30·2	26·6	185·6	1·75	0·33	58·2	50·0	2·13	66·13
B. Malabarica (expressed oil)	0·8586	32·0	12·1	187·2-188·5	1·35	0·55	63·6	49·5	2·55	58·56
B. Payena oleifera	0·8685	29·1	54·0	183·9-185·0	1·86	0·50	58·6	52·5	2·03	50·79

Constants of Insoluble Fatty Acids of Bassia Oils.

Name.	Per Cent.	Melting-Point, ° C.	Iodine Value.	Neutralisation Value.	Mean Molecular Weight.	Liquid Acids, per Cent.	Melting-Point of Liquid Acids.	Iodine Value of Liquid Acids.	Neutralisation Value of Liquid Acids.	Mean Molecular Weight of Liquid Acids.	Stearic Acid in Total A per Cent.	Melting-Point of Stearic
B. butyracea ...	95·08	53·3	38·4	195·9	281·9	—	—	—	—	—	nil	—
B. latifolia	93·95	48·3	48·5	189·9	295·2	59·5	34·4	81·1	177·6	315·7	13-25	66·9
B. longifolia ...	93·70	45·6	52·1	188·7	297·1	20·5	30·2	87·3	173·8	322·7	12-20	67·2
B. Malabarica (expressed oil) ...	94·00	45·8	59·5	192·1	291·9	—	—	—	—	—	—	—
B. Payena oleifera	91·65	40·0	59·0	192·2	291·7	—	—	—	—	—	nil	—

The following expressed seed oils have also been examined. The oil from the seeds of *S. trifoliatus* was, however, obtained by ether extraction.

	Jatropha glanduifera.	*Luffa acutangula.*	*Mimusops elengi.*	*Pithecolobium dulce.*	*Psoralea corylifolia.*	*Sapindus trifoliatus.*	*Thespesia populnea.*	*Vernonia anthelmintica.*
FAT:								
Acid value	15·79	93·7	45·5	63·9	39·2	42·7	48·1	58·2
Saponification value ...	194·5	229·2	213·9	205·9	204·6	191·8	201·4 to 204·0	202·9
Reichert-Wollny value ...	4·0	13·1	10·6	8·4	6·9	1·61	5·9	7·9
Insoluble volatile acids, titration number ...	0·41	0·83	0·68	0·34	0·71	0·37	0·55	0·35
Unsaponifiable matter, per cent.	1·38	1·09	1·56	1·17	1·73	1·10	—	1·79
Butyro - refractometer at 25° C.	76·5	73·0	73·5	69·5	79·5	74·0	81·5	75·0
Sp. gr. 100°/15° C. ...	—	0·9002	0·8777	0·8756	0·8756	0·8213	0·8670	0·8835
Iodine value	—	40·1	66·5	56·6	79·9	58·6	71·5	71·0
FATTY ACIDS (INSOLUBLE):								
Per cent.	89·01	82·3	89·4	87·64	88·3	93·9	93·5	91·6
Melting-point, ° C. ...	35	44·1	35	44·7	32·8	54·4	43·9	35·6
Iodine value	119·6	41·9	68·1	57·6	83·1	57·0	75·3	73·4
Neutralisation value ...	187·3	215·0	202·1	198·7	192·4	188·6	196·9	195·1
Mean molecular weight ...	299·4	260·9	277·6	282·2	291·5	297·3	284·7	287·4

A. R. T.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of the Enzymatic Activity of Nuclease. G. Pighini. (*Zeitsch. physiol. Chem.*, 1910, **70**, 85-93.)—The method proposed depends on the fact that the nuclease contained in blood-serum acts on nucleic acid, with the result that the optical rotation of the latter substance is gradually destroyed. Two c.c. of the serum are added to a quantity of nucleic acid solution sufficient to fill the polarimeter tube (say 22 c.c.), and the optical rotation of the mixture is observed; the mixture is then maintained at a temperature of 37° C., and the observations are repeated at intervals of an hour or so. For comparative estimations the proportion of the serum to the nucleic acid must be the same in each test, and it will be found most convenient to heat the mixtures in the polarimeter tubes, a number of the latter being employed. The nucleic acid solution is prepared by dissolving 1·60 grms. of nucleic acid in 100 c.c. of a 0·85 per cent. sodium chloride solution and adding 12 drops of ammonia. Results of experiments recorded show that the action of the nuclease is continuous, and that it proceeds most rapidly during the first hour; the rotation of the nucleic acid is destroyed completely in from twelve to twenty-four hours. After being heated to a temperature of 65° C., nuclease no longer possesses enzymatic properties.

W. P. S.

Method for Ascertaining whether a Liquid Contains One or More Enzymes. Achalme and Bresson. (*Comptes rend.*, 1910, **151**, 1369-1372.)—When a fermentative extract is obtained which acts on several substances, it is difficult sometimes to determine whether its multiple activity is to be ascribed to one and the same enzyme or to the presence of a plurality of enzymes in the same extract. The authors have devised a method for solving this question, which depends on the following hypothesis: If a limited quantity of a single enzyme be caused to act simultaneously on two different substrata, its action will be divided between the two. But if the liquid contain two enzymes, each will act on its own specific substratum independently of the other. Accordingly, the mode of procedure is as follows: Under identical conditions of temperature, concentration, acidity, or neutrality, a sufficiently small quantity of the active liquid to permit of the action being followed easily from hour to hour is caused to react upon solutions of each substance separately, and on a mixture of the two. If the action on the mixed substances represents approximately the sum of the action on the two separately, it is to be inferred that two distinct enzymes are concerned. If, on the other hand, the action on the mixed substances does not appreciably exceed that on the separate substances, only one enzyme is at work. Instead of estimating the quantity of substance modified in a given time, it may be convenient to determine the time necessary for complete hydrolysis. In the case of two enzymes this time will be practically the same for all three solutions, whereas in the case of a single enzyme the time is much longer in the liquid containing two substances to be converted. The authors have proved their hypothesis in the cases of emulsin and invertase.

J. F. B.

Method of Destroying Organic Matters in Testing for Mineral Poisons. E. Jungfleisch. (*Comptes rend.*, 1911, **152**, 199-200.)—The following process is recommended for completely destroying organic matters previous to testing for the presence of mineral poisons in animal organs, etc. About 300 grms. of the material are heated in a flask with about 300 c.c. of concentrated sulphuric acid while a current of nitrous vapours is passed through the mixture. The nitrous vapours are most conveniently obtained by passing a current of sulphur dioxide (from a siphon containing liquid sulphur dioxide) through a wash-bottle containing about 500 c.c. of concentrated nitric acid; a tube leading from the wash-bottle conducts the vapours to the bottom of the digestion flask. The nitrous vapours act catalytically in the destruction of the carbonaceous matters, and a perfectly colourless solution is obtained after about four hours' heating. The quantity of nitric acid mentioned is sufficient for the destruction of four or five quantities of 300 grms. of organic substance. W. P. S.

Physical Constants and Analyses of Certain Commercial Peptones. G. Pépin. (*Bull. d. Sciences Pharmacol.*, 1910, **17**, 594-599; through *Chem. Zentralbl.*, 1910, **2**, 1932-1933.)—The eleven preparations experimented with are classified in three groups according to their method of manufacture—(1) by the action of trypsin on flesh or fibrin, or by the action of pepsin on (2) flesh or fibrin or on (3) gelatine. The percentage of nitrogen when calculated to an ash-free basis lay between 12·6 and 17·6. The ratio of amino-nitrogen to total nitrogen varied between 1 : 4 and 1 : 36. The amino-nitrogen was much higher in the tryptic peptones than in the peptic peptones; and, of the latter, those derived from gelatine were poorer in amino-nitrogen than those derived from flesh or fibrin. The ash content was highest in the peptic peptones from flesh, in some as high as 20 per cent., and lowest in the tryptic products. The ash of the peptic products consists mainly of the chlorides of sodium and calcium, whilst carbonates are present in large amount in that of the tryptic peptones. The peptic peptones rarely contain phosphorus, magnesium, or iron, and then only in traces. The large quantities of calcium found in some of them was doubtless due to lime or chalk used for purposes of neutralisation.

For the purpose of determining the physical constants, solutions were made of such strength that each contained precisely 1 per cent. of nitrogen. The tryptic peptones had the greatest effect in depressing the freezing-point, the peptic peptones from gelatine having the least. The greatest optical activity is shown by the peptic peptones from gelatine, the least by the tryptic products. The electrical conductivity of the peptic products derived from flesh and fibrin was greater than that of the other preparations. G. C. J.

Microchemical Detection of Protein. T. Bokorny. (*Chem Zeit.*, 1911, **35**, 69-70.)—The author has based a microchemical method for the detection of protein on the coagulation phenomena and the formation of "proteosomes," observed when the subepidermal cells of certain plants of the order Crassulaceæ are treated with a solution of caffeine. The formation of these proteosomes is observed in the plasma of the cells in superficial sections of the leaf, generally in the form of closely crowded bodies of angular shape, or, if not so numerous, of a flattened, rounded shape. After contraction of the vacuole wall by the action of 10 per cent. nitric acid, or better of the caffeine solution itself, they are at liberty to assume a spherical form. The proteosomes are very lustrous and perfectly clear; their formation takes place distinctly in the plasma, and in many cases in the cell sap itself. Certain observers have mistaken them for a precipitate of caffeine tannate. The coagulation of the proteosomes may conveniently be studied with *Spirogyra*. The objects are treated with a 0·5 per cent. solution of caffeine, and the tube is suspended for a few minutes in a water-bath at 60° C. The coagulated proteosomes then appear very cloudy; on treatment with absolute alcohol they remain intact in the cells. The coagulation of the proteosomes prior to treatment with alcohol is recommended, because freshly-formed proteosomes are dehydrated so suddenly by alcohol that certain observers have stated that they are soluble. The proteosomes also undergo gradual coagulation on keeping for four days at the ordinary temperature in a saturated solution of caffeine. They may also be coagulated by chemical reagents—*e.g.*, a solution containing 8 per cent. of acetic acid, 4 per cent. of sodium chloride, and 0·5 per cent. of caffeine; the reagent acts very rapidly, the proteosomes lose their lustre and become opaque. J. F. B.

ORGANIC ANALYSIS.

Use of Calcium Carbide for Determining Moisture. I. Masson. (*Chem. News*, 1911, **103**, 37-38.)—The use of calcium carbide affords a reliable means for determining water in many substances, and especially in organic materials, and it is chiefly of value in those cases in which the material cannot be heated without giving off water of decomposition, or evolving vapours other than water-vapour, or without gaining weight by oxidation.

Calcium carbide in these cases, on being mixed with the substance, gives off acetylene readily, which may be measured. The method of Danne (1900) was to treat the powdered material in a test-tube with a layer of sand and an upper layer of carbide, and to heat the material, when the water-vapours acted on the carbide, with the evolution of acetylene, which was collected, the apparatus being standardised with known amounts of water. Dupré (ANALYST, 1906, **31**, 213) proposed a similar method for determining moisture in cordite and other explosives. It has been shown, however, that it is unnecessary to heat materials in this way, simple intimate admixture of the powdered carbide (in large excess) and the material being sufficient to bring about complete reaction. Such a method is now employed in Australia in estimating the moisture in sheep's wool (O. Masson, *Proc. Soc. Chem. Ind.* [Victoria], 1909). A known weight of wool clippings is introduced into a metal globe, which has a screw top and a tray in its interior, below the tray being the powdered carbide in considerable excess. A gas exit-tube is also provided. On shaking, the carbide falls on to the wool, and the reaction proceeds rapidly. The increased pressure obtained, the volume being kept constant, is observed by means of a mercury manometer suitably graduated.

The carbide method has been used by Roberts and Fraser (ANALYST, 1910, **35**, 217) in the case of hydrous petroleum products, these being rendered fluid, if necessary, by the addition of dry paraffin oil. The acetylene is measured by the quantity of brine displaced from a bottle, the necessary temperature correction being made.

With regard to the determination, by the carbide method, of water of crystallisation, the author finds (*J. Chem. Soc.*, 1910, **97**, 351) that hydrated salts may be divided into four classes, containing (1) those substances, like $Na_2CO_3.10H_2O$, which react completely; (2) those, like $BaCl_2.2H_2O$, which react only on heating; (3) substances which only react partially either in the cold or on heating ($CuSO_4.5H_2O$); (4) substances which are not affected by carbide even at 170° C.

The method used for wool is likely to be found useful in the case of materials like cotton, paper, fabrics, wood-pulp, tobacco, and flour. Modifications of the carbide method may also be employed for the examination of soils, minerals, guano, and coal. Substances like butter, cocoa, etc., may be mixed with a fat solvent like benzene to facilitate the action of the carbide. In all cases standardisation with known quantities of water is necessary; the yield of acetylene is found to be practically constant at 10·5 c.c., at 0° C. and 760 mm., for every 18·0 mgrms. of water (theory = 11·2 c.c.). A. R. T.

Direct Estimation of Caoutchouc in Vulcanised Rubber Articles. G. Hübener. (*Chem. Zeit.*, 1911, **35**, 113-115.)—Continuing the development of his bromination method (ANALYST, 1909, **34**, 170, 365; 1910, **35**, 266), the author has investigated further cases of admixtures which may possibly influence the validity of the results. In the present paper the effect of the presence of bituminous bodies, asphalt and "mineral rubber," has been dealt with, and it has been found that such bodies, under the prescribed conditions of bromination, yield insoluble bromo derivatives which vitiate the results by increasing the apparent percentage of caoutchouc. In presence of such admixtures a modification of the original method is necessary, which consists in eliminating the bituminous bromo derivatives by extraction, after bromination and before the titration of the combined bromine. The procedure is as follows: 0·1 grm. of the sample is brominated in the prescribed manner, and the mixed bromides are collected on a filter and washed with hot water. The funnel is then removed to another flask, and the precipitate is dehydrated by washing with absolute alcohol. It is then transferred to a beaker, in which it is digested at a moderate temperature with a mixture of equal parts of carbon tetrachloride and alcohol. The extract is decanted off, and the digestion is repeated until the liquid remains colourless. The precipitate is then filtered off, and the carbon tetrachloride is eliminated by washing with absolute alcohol. The filter and precipitate are placed in an Erlenmeyer flask with 20 c.c. of $\frac{N}{10}$ silver nitrate and 25 c.c. of strong nitric acid; the liquid is heated at first gently, until the violent reaction due to the presence of the alcohol has subsided, and is finally evaporated to a volume of about 15 c.c. The excess of silver is then titrated in the usual manner.

The author has proved, by the analysis of mixtures of known composition, that the bromides of bituminous matters are satisfactorily eliminated by the above treatment, whilst the caoutchouc bromide is quite unaffected. Further experiments have shown that when rubbers other than Para are present, containing resins soluble in acetone, the bromination method shows with satisfactory accuracy the percentage of ue caoutchouc, independent of the resins of the crude rubber. J. F. B.

New Test for Cupreïne. G. Denigès. (*Comptes rend.*, 1910, **151**, 1354-1355.)—Cupreïne occupies a position among the quino-cinchonine alkaloids analogous to that of morphine among the morpholic group—*i.e.*, it possesses a phenolic function.

It therefore yields a characteristic colour reaction with the same reagents as have been proposed by the author as a test for morphine. Ten c.c. of a 0·2 per cent. solution of cupreïne sulphate are placed in a test-tube, 1 c.c. of ammonia, and 1 c.c. of hydrogen peroxide (of a concentration not exceeding 1 volume), are added. After shaking, 0·1 c.c. of a 3 to 4 per cent. solution of copper sulphate is introduced. A fine blue coloration rapidly develops in the liquid, which gradually becomes darker and cloudy, holding in suspension greenish-blue corpuscles which ultimately settle to the bottom. The liquid becomes clear, with a fine emerald-green coloration, if it be mixed with an equal volume of alcohol, or if it be acidified with acetic or hydrochloric acid. The addition of a large excess of the latter acid or of sulphuric acid turns the colour to yellowish-red. The green solution shows an absorption band in the neighbourhood of the infra-red. The test is very delicate, and is perceptible with a solution containing 0·02 to 0·03 grm. of cupreïne sulphate per litre, if it be performed against a blank; no precipitate, however, is formed at that dilution. The test may be applied to the alkaloid *in situ*—*e.g.*, in the tissues of the plant—by moistening with a drop of a mixture of 1 c.c. of a 3 to 4 per cent. solution of copper sulphate, 5 c.c. of ammonia, and 10 c.c. of water, then touching the objects with a drop of hydrogen peroxide of not more than one-half volume strength. J. F. B.

Estimation of Formaldehyde. F. Herrmann. (*Chem. Zeit.*, 1911, **35**, 25-26.)—The method of estimating formaldehyde in the form of hexamethylenetetramine may be rendered accurate by causing the ammonia to be liberated from ammonium chloride within the liquid itself. Under these conditions the heat of the reaction causes the nascent ammonia to react instantaneously and quantitatively with the formaldehyde. From 4 to 4½ grms. of the formaldehyde solution are shaken with 3 grms. of pure powdered ammonium chloride in a stoppered flask, into which is subsequently introduced, as rapidly as possible and with constant shaking, 25 c.c. of 2N-sodium hydroxide solution. After the contents of the flask have cooled down to the ordinary temperature, they are diluted with 50 c.c. of water, and titrated with N-sulphuric acid, with methyl orange as indicator. The number of c.c. of N-sodium hydroxide solution consumed in the formation of the hexamethylenetetramine, multiplied by the factor 0·06 gives the amount of formaldehyde (in grms.) in the solution under examination. A correction must be applied for the amount of free acid usually present in commercial formaldehyde solutions. The results thus obtained in the duplicate estimation quoted were closely concordant.

C. A. M.

Separation of Liquid from Solid Fatty Acids—II. P. Falciola. (*Gazz. Chim. Ital.*, 1910, **40**, 425-435.)—Further experiments upon the method of separating liquid from solid fatty acids in natural fats by the method already described (ANALYST, 1911, 23) have shown that the solubility of ammonium stearate and palmitate in absolute alcohol is increased by the presence of ammonium oleate. The following results illustrate the degree of accuracy obtainable in the separation of these fatty acids from a larger quantity of oleic acid: A mixture of 5 grms. of oleic acid, 2·5 grms. of palmitic acid, and 2·5 grms. of stearic acid, was treated with the ammoniacal alcohol at 0° C., as previously described, and the separated oleate was found to contain 0·20 grm. of ammonium palmitate and 0·04 grm. of ammonium stearate. In the case of a mixture of the fatty acids melting at 48·2° C., and having an iodine value of 30·1, the solid fatty acids separated by this method melted at 54·5° to 56° C., and had an iodine value of 6·6 to 8. The method failed to give good results with certain oils, although in the case of sesame oil it effected a separation of the fatty acids into solid acids that did not give Baudouin's reaction, and liquid fatty acids which gave a pronounced reaction. Olive oil, however, left no insoluble ammonium salts upon the filter. Pure ammonium salts of other fatty acids were prepared, and their solubility in different solvents ascertained. *Ammonium linolate* is a soap-like salt which begins to melt at 57° to 58° C. It is readily soluble in methyl alcohol, and dissolves to the extent of 35 per cent. in ammoniacal absolute alcohol at 0° C. It is sparingly soluble in ether, but is soluble in hot benzene and acetone, and in cold carbon tetrachloride and chloroform. *Ammonium erucate* (melting-point 70° to 77° C.) is soluble in methyl and ethyl alcohols, sparingly soluble in ether, benzene, and chloroform, and nearly insoluble in cold acetone and carbon tetrachloride. *Ammonium laurate* (melting-point about 75° C.) is insoluble in cold water. Absolute alcohol at 7° C. dissolves about 4·8 per cent., whilst ammoniacal alcohol at the same temperature dissolves 3·5 per cent. It is soluble in methyl alcohol and in hot benzene, from which it crystallises on cooling, but is only sparingly soluble in ether and acetone. *Ammonium myristate* (melts imperfectly at 75° C.) is sparingly soluble in cold water, cold acetone and methyl alcohol, but is somewhat more soluble in alcohol. It is nearly insoluble in cold ether and carbon tetrachloride. *Ammonium caprylate* (melting-point 70° to 85° C.) forms a turbid solution in water. It is soluble in methyl and ethyl alcohol and in chloroform, and sparingly soluble in ether, carbon bisulphide, and chloroform. It may be obtained in crystalline form by cooling its solution in hot benzene. *Ammonium crotonate* forms white crystals melting at 105° to 115° C. It is readily soluble in cold water, methyl alcohol, and alcohol, and sparingly soluble in ether, carbon tetrachloride, benzene, and acetone. *Ammonium butyrate* is a deliquescent salt melting between 70° and 85° C. It is very soluble in alcohol, methyl alcohol, and chloroform, but is nearly insoluble in ether, acetone, or benzene. C. A. M.

Estimation of the Total Fatty Acids in Fat, Oils, and Soaps. P. Simmich. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 38-44.)—The author considers that the only accurate method of estimating the total fatty acids in oils, etc., consists in separating the fatty acids (liberated in the usual way from the saponified oil) by means of a mixture of ether and petroleum spirit, evaporating the solvent in a current of carbon dioxide, and exactly neutralising the residue of fatty acids by the addition of $\frac{N}{2}$ potassium hydroxide solution (alcoholic). The resulting soap is then dried, firstly in an atmosphere of hydrogen and then under reduced pressure at a temperature of 100° C., and weighed. The quantity of fatty acids is obtained by subtracting from the weight of the soap 0·01907 grm. for every c.c. of $\frac{N}{2}$ potassium hydroxide solution required for the neutralisation. Incorrect results are obtained if the fatty acids be dried in their free state before being weighed. W. P. S.

Characteristics of Neutralised Olive-Residuum Oils. L. Archbutt. (*J. Soc. Chem. Ind.*, 1911, **30**, 5-6.)—During the last few years products derived from the residual marc and the stones of the olives left after expression of ordinary olive oil have been sold for lubricating purposes under the name of "neutral" or "saponified" oils. The free acids which are originally present in these are removed by treatment with alkali and washing with water, so that a product with a guaranteed maximum acidity not exceeding 1 to 4 per cent. is obtained. These oils are usually turbid and often of a dark colour, and as a rule they contain moisture due to imperfect drying after the final washing. They frequently contain solid fat in sufficient quantity to cause them to give abnormally high results in Redwood's viscometer. They are also peculiar in their high proportion of unsaponifiable matter, which may exceed 3 per cent., and cause them to yield a precipitate in Bellier's test for arachis oil. Hence Renard's process should also be used before drawing a conclusion as to the presence of arachis oil. These oils also give an abnormal coloration (an immediate brown or greenish-brown emulsion) when shaken with an equal volume of nitric acid of sp. gr. 1·375, although the colour is quite distinct from that obtained with cottonseed oil. With Halphen's reagent no coloration is obtained. They oxidise more readily than neat's-foot oil or lard, but, in the author's opinion, are quite suitable for lubrication when used in admixture with a mineral lubricating oil. The following results were obtained with commercial samples:

Sp. gr. at 15·5° C. ...	0·9167	0·9165	0·9175	0·9186	0·9169	0·9175	0·9169
Efflux velocity of 50 c.c. from viscometer at 15·5° C.* (seconds) ...	516	450	437	478	480	561	524
Saponification value ...	186·10	186·90	189·30	186·90	186·00	185·50	185·90
Iodine value	86·40	85·10	85·80	84·40	85·50	85·60	85·00
Free acid (oleic), per cent.	2·90	3·30	1·00	1·10	0·90	2·30	1·80
Unsaponifiable matter, per cent.	2·49	2·34	2·08	2·70	3·30	3·32	3·23

* Normal olive oil = about 435 to 446 seconds.

C. A. M.

Analysis of Gelatins. J. Herold jun. (*Chem. Zeit.*, 1911, **35**, 93-94.)—The difference between gelatin and glue may be said to lie in the relative proportions of glutin and glutose in each. Glutin is the gelatinising constituent of gelatin, and the best gelatin is that which contains the highest proportion of it. On the other hand, the presence of glutin in glue is actually a drawback to its adhesive power, and a high proportion of non-gelatinising glutose is the main desideratum. Glutose may be prepared from glutin by the regulated action of alkalies, or merely by prolonged heating with water. The best method of preparing glutose is by heating a solution of gelatin with strong ammonia in a sealed tube for two hours at 100° C., and removing the ammonia, after filtration, by evaporation. The author has previously described a method for the determination of the melting-points of gelatin jellies (ANALYST, 1910, **35**, 174). By mixing glutose and gelatin, he has observed that the melting-point of a 20 per cent. jelly is lowered in proportion to the amount of glutose present. For reasons explained (*loc. cit.*), the absolute melting-point of the jelly has no analytical significance, but the differences between the melting-points of jellies from the same gelatin at different concentrations may form the basis of a method of valuation. Preliminary observations with the purest form of gelatin procurable showed that the difference between the melting-points of 20 and 10 per cent. jellies was 1·2° C. This sample of gelatin contained 17 per cent. of moisture and 1 per cent. of ash; hence glutin = 82 per cent. by difference. If now the difference between the melting-points of 20 and 10 per cent. jellies of any other sample of gelatin be found to be $a°$ C., the percentage of glutin in the latter sample may be calculated by the expression, $\frac{a}{1{\cdot}2} = \frac{x}{82}$. The percentage of glutose will then be 100 – (water + ash + glutin).

This method gives results in complete accordance with the commercial valuation of the gelatin, but it requires the accurate determination of the melting-points to within 0·1° C., under strictly standard conditions. The author describes an apparatus for this purpose, consisting of a glass tube surrounding the lower part of the thermometer, leaving an annular space of 1 mm. The tube is closed at the bottom by a plug and a globule of mercury. When the jelly has been set around the thermometer (*e.g.*, at 19° C. for half an hour), the plug and mercury are removed; the temperature is slowly raised in a small glass air-bath surrounded by water at 5° C., and when the tube begins to slide down the thermometer the melting-point is observed. Since the difference in melting-point is strictly proportional to the concentration of the jelly, determinations may be made with 30 and 20 per cent. jellies if the sample be a very weak one. J. F. B.

Quantitative Estimation of Active Hydrogen in Organic Compounds by Means of Magnesium-Methyl Iodide. T. Zerewitinoff. (*Ber.*, 1910, **43**, 3590-3595.)—In previous papers (ANALYST, 1907, **32**, 299; 1908, **33**, 366) the author has described his gasometric method for the estimation of groups containing active hydrogen—*e.g.*, OH, SH, NH, and NH_2 groups—in organic compounds. This method has proved of service, even in the case of such complicated compounds as sugar and flavone, and sufficiently accurate results are generally obtained. In the present paper the author deals with the alkaloids—derivatives of tertiary amines. These react with magnesium-methyl iodide in pyridine solution if they contain hydroxyl groups, and the results at the ordinary temperature are practically identical with those obtained on heating to 85° C.; they therefore contain no amino group. The author has also studied certain tautomeric compounds, containing the group $CO.CH_2$, which is capable of assuming the form C(OH) : CH. These yield results somewhat below the theoretical value at the ordinary temperature, but give accurate values at 100° C. Similarly, primary and secondary nitro compounds of the aliphatic series are capable of assuming a pseudo-acid form, in which they form salts in virtue of the labile group $\mathrm{R.CH : N}\genfrac{}{}{0pt}{}{/\!\!/\mathrm{O}}{\backslash\mathrm{OH}}$, which reacts with magnesium-methyl iodide. These also give results somewhat lower than the theoretical at the ordinary temperature, but at 100° C. the values more nearly approach the theoretical, the conversion into the isomerides, however, never being quite complete. The author has investigated the applicability of other solvents than pyridine; these must be inactive towards the magnesium-methyl iodide, and preference is given to those of high boiling-point, for work at higher temperatures, and those having such a small vapour pressure that a correction is unnecessary. Suitable solvents are: Amyl ether (already proposed by Sudborough and Hibbert), anisol, and the hydrocarbons xylene and mesitylene. The three last solvents were distilled from sodium; the magnesium-methyl iodide was always prepared in amyl ether solution. These all gave satisfactory results with hydroxyl and amino groups. As a rule the amino groups only react with one hydrogen atom at the ordinary temperature, but on heating the mixture up to 85° C. in five minutes, the second hydrogen atom reacts quantitatively. J. F. B.

Estimation of Iodine in Organic Compounds and its Separation from other Halogens. A. F. Seeker and W. E. Mathewson. (*U.S. Dept. of Agric., Bureau of Chem.*, Circular No. 65, 1910, 1-5.)—The following process was found to yield reliable results when applied to the estimation of iodine in such compounds as erythrosine, iodoform, and phenyl iodide; it depends on the fact that the organic matter is completely oxidised when the substance is treated with potassium permanganate and nitric acid, the chlorine and bromine being converted into volatile products, whilst the iodine remains as iodic acid. A weighed quantity of about 0·4 grm. of the erythrosine is placed in a porcelain basin and dissolved in 5 c.c. of a 10 per cent. sodium hydroxide solution; 35 c.c. of a 7 per cent. potassium permanganate solution are then added, and, when the basin has been covered, 10 c.c. of nitric acid are introduced. The mixture is heated on a water-bath, the cover is removed when effervescence has ceased, and the solution is evaporated to dryness. A further quantity of 5 c.c. of concentrated nitric acid and 5 c.c. of the permanganate solution are added to the residue, and the evaporation is repeated. The residue is now taken up with 50 c.c. of water and 5 c.c. of nitric acid, 40 c.c. of a saturated sulphur dioxide solution are added, and, when the manganese hydroxide has dissolved, the iodine is precipitated by the addition of silver nitrate. After the mixture has been boiled to remove the excess of sulphur dioxide, the silver iodide is collected and weighed. In the case of volatile compounds like phenyl iodide the oxidation should be carried out in a flask attached to a reflux condenser. The Carius method was found to yield erratic and unreliable results when employed for the estimation of iodine in erythrosine. W. P. S.

Methyl Red as an Indicator compared with Methyl Orange. H. T. Tizard. (*J. Chem. Soc.*, 1910, **97**, 2477-2490.)—The methyl red of Rupp and Loose (*Ber.*, 1908, **41**, 3905), which has the constitution $NMe_2.C_6H_4.N:N.C_6H_4.CO_2H(o)$, is greatly superior to methyl orange as an indicator. The visible change, red to yellow, takes place between $10^{-5\cdot7}$ and 10^{-7}—that is, between H· concentrations $\frac{N}{200,000}$ and $\frac{N}{10,000,000}$. Not only, therefore, is the end-point very much sharper than when methyl orange is used, but the neutral point so found is very much nearer the theoretical neutral point. Methyl red is especially valuable for the exact titration of moderately weak bases, such as ammonia, by strong acids.

The following method of preparation is given: Five grms. of anthranilic acid are dissolved in 150 c.c. of water and 15 c.c. of concentrated hydrochloric acid. To this 2·5 grms. of solid sodium nitrite are added, and the solution kept for half an hour. It is then poured into a solution of 4·65 grms. dimethyl-aniline in a mixture of 5 c.c. of concentrated hydrochloric acid and 50 c.c. of water. Fifty grms. of sodium acetate are added, and the mixture is warmed to 40° C., when the red product begins to separate at once, though the reaction is not complete for some three hours. The substance is then collected, and can be recrystallised from glacial acetic acid. The yield is almost quantitative. Methyl red is very insoluble in water. Its saturated solution at ordinary temperature is only about $\frac{N}{100,000}$. It is, however, easily soluble both in acids and in alkalis. G. C. J.

Analytical Constants of Shellac, Lac-Resin, and Lac-Wax. Puran Singh. (*J. Soc. Chem. Ind.*, 1910, **29**, 1435-1436.)—The following constants were determined by the author on samples of pure shellac (free from orpiment and colophony) prepared by himself from three varieties of crude lac obtained from the Forest Department, Central Provinces, India. For comparison, the figures yielded by a factory sample are included, and this sample contained about 0·3 per cent. of "yellow arsenic" (orpiment). The constants of lac-wax and lac-resin were also determined:

Variety.	Moisture at 100° C.	Matter insoluble in Hot Alcohol.	Acid Number (A).	Saponification Number (B).	Ester Number (B-A).	Iodine Absorption (Hübl). 18 hours.	Endemann Number.
	Per Cent.	Per Cent.				Per Cent.	
Shellac from Kusum lac (from *Schleichera trijuga*)	2·7	0·7	61·1	201·0	139·9	9·6	8·4
Shellac from Palas lac (from *Butea frondosa*)	3·8	0·8	60·8	202·4	141·6	9·3	8·0
Shellac from block lac (*i.e.*, lac compressed during transit)	3·9	1·1	63·1	201·6	138·5	8·2	9·2
Mirzapur factory shellac ...	2·0	0·6	64·4	203·6	139·2	8·6	7·4
Lac-wax (melting-point 58°-59° C.)	—	—	21·1-24·3	79·2-85·0	57·1-60·7	8·8	—
Lac-resin (desiccator dry)	—	—	52·1-59·2	193·5-198·4	139·2-141·4	6·8-7·3	7·3-8·1
Lac-resin (melted)... ...	—	—	54·9	190·0	135·1	5·9	—

The three shellacs were prepared by extraction with wood-spirit, and not by repeated melting, so that they were free from the injurious effects of overheating. The "Endemann number," which is nearly constant for the best qualities of shellac at about 7 to 8 per cent., was proposed by Endemann in 1907 (*J. Franklin Inst.*, 1907, **164**, 285; 1908, **165**, 217; *cf.* ANALYST, 1907, **32**, 425) as a method for shellac examination, and consists in dehydrating 1·5 to 2 grms. of the sample by heating with 3 to 4 grms. of sand and strong hydrochloric acid. The author modifies the method by first heating the shellac for two hours at 100° to 105° C. with calcined precipitated silica, and then adding hydrochloric acid. The mixture is evaporated until thoroughly dry, and then extracted completely with alcohol. The proportion of non-condensible resin and fatty acids thus obtained is stated not to exceed 8 per cent. in genuine samples. A. R. T.

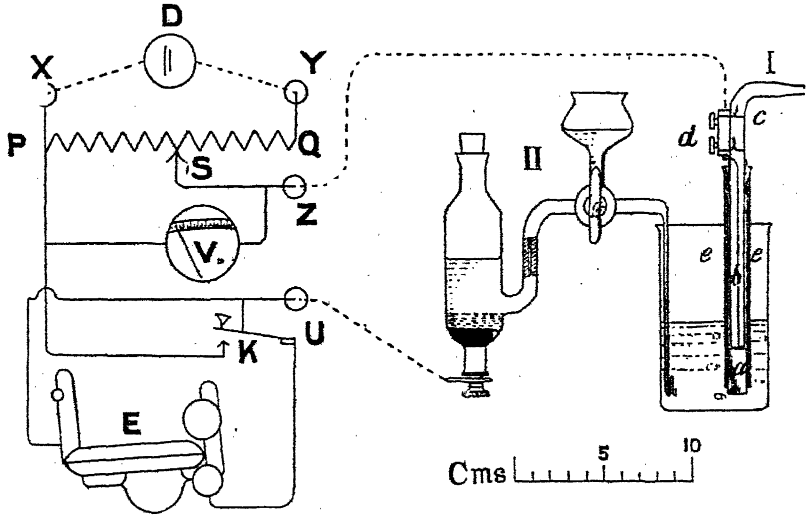
Note on the Composition of Soot. H. W. Harvey. (*J. Agric. Sci.*, 1910, **3**, 398-399.)—Samples of soot when analysed by Kjeldahl's method show from 0·5 to 11 per cent. of nitrogen, which is present mainly as ammonium chloride, though usually stated to exist as ammonium sulphate. The following table shows the relation between the nitrogen content and the weight per bushel:

Type of Soot.	Percentage of Nitrogen.	Pounds of Soot per Bushel loosely packed.	Pounds of Nitrogen per Bushel of Soot.
From kitchen chimney	11·0	9	1·0
" " "	5·4	23	1·2
From coal and wood fire	4·5	26	1·2
From dwelling houses (mixed)	3·6	29	1·0
" " " "	2·9	28	0·8
From 40-foot boiler shaft	0·5	47	0·23

The author concludes that soot should be bought by volume and not by weight, thus insuring an approximately constant quantity of nitrogen per bushel. Farmers generally are of opinion that a light soot is the best, and the figures quoted confirm this. There is some loss of nitrogen when soot is allowed to stand in a loose heap, as in the case of the mixed sample. H. F. E. H.

Determination of Sucrose in Beet Sugar Factory Products by Clerget's Process, using Invertase as Hydrolyst. J. P. Ogilvie. (*J. Soc. Chem. Ind.*, 1911, **30**, 62-64.)—In preparing the sugar solution for inversion by invertase, it is necessary to avoid the presence of acids, alkalis, or other reagents capable of inhibiting the action of the enzyme. In the case of highly-coloured sugar-factory products some special process of clarification must therefore be employed. The author describes the following method: Four times the normal weight of the sample (*e.g.*, molasses) are placed in a 200 c.c. flask, defecated with the minimum quantity of basic lead acetate and a little alumina cream, diluted to the mark, shaken and filtered. One hundred c.c. of the filtrate are treated with a current of sulphur dioxide gas until a faint odour persists. The liquid is transferred to a 200 c.c. flask, diluted to the mark, shaken and treated with a little dry chalk and kieselguhr before filtration. In this way a normal clarified solution is obtained which is neutral and free from lead. For the inversion, 50 c.c. are placed in a 100 c.c. flask and heated in a thermostat from 50° to 55° C.; 0·5 grm. of washed brewery yeast are added and 2 drops of acetic acid. The liquid is digested at the above temperature for four and a half to five hours, diluted to 100 c.c. with the addition of alumina and kieselguhr, filtered, and polarised. With pure sugar the Clerget constant obtained in this way was 131·6, as against 132 for the acid method of inversion. With beet molasses, however, the invertase method gave appreciably higher results than the acid method. This discrepancy was found to be due to observing the direct polarisation in alkaline solution and the polarisation after inversion in acid solution, when the ordinary acid method of inversion is used. The author has proved that if both polarisations be observed in acid solution, the acid method yields practically the same results as the invertase method. The most convenient modification of the acid method is that described by Pellet: The molasses solution is defecated by basic lead acetate and filtered. A portion of the filtrate is treated with a slight excess of sulphurous acid solution, to precipitate the lead, and filtered. The acid filtrate thus obtained is used for the direct polarisation and, with the addition of hydrochloric acid, for the inversion. J. F. B.

Electrometric Method for the Estimation of the Acidity of Tan Liquors. H. J. Sand and D. J. Law. (*J. Soc. Chem. Ind.*, 1911, **30**, 3-5.)—Owing to the difficulty of finding indicators that will give a sharp change of colour in dark tan liquors, the authors have devised an electrometric method of estimating the original hydrion concentration of the liquid, and the amount of alkali which must be added until there is reached a hydrion concentration corresponding to the change of colour with some indicator, such as phenolphthaleïn. In their opinion there is a relationship between the former and the swelling power of the liquid, and between the latter and its lime-solvent capacity. The method is based upon Böttger's observation (*Zeitsch. phys. Chem.*, 1897, **24**, 253) that when metals and also hydrogen absorbed by platinum black are brought in contact with solutions containing the same substances in the form of ions, a potential difference, depending upon the nature of the substances and the concentration of the ions, is established. The apparatus used by the authors for the titration of tan-liquors consists of the hydrogen electrode I. and the auxiliary electrode II., both of which dip into the liquid, and the potentiometer



box for the measurement of the difference of potential between them. These electrodes are represented on the right of the diagram, whilst the electrical apparatus is shown on the left. The hydrogen electrode I. consists of a thin platinum plate, *a*, attached to a long thin platinum wire, *b*, which is fitted into the glass tube *c*, and, passing through the sealed end of the tube, ends in the terminal *d*. A glass tube, *e*, connected with *c* by means of rubber, surrounds the plate *a* to within about 2 mm. from the bottom, and the plate itself is coated with platinum black. Hydrogen, purified by passage through permanganate solution, enters at the tube *c*, and escapes at the bottom, from twenty-five to thirty minutes being required to complete the saturation of the platinum black with the gas. Subsequently only about one minute's passage of hydrogen is required to repair the loss in saturation caused by the transference of the electrode from one liquid to the other before proceeding with the titration. The auxiliary electrode vessel II. has a normal calomel electrode, and the left limb of the capillary connection is filled from the funnel with N-potassium chloride solution, the other limb being charged with a $\frac{3}{5}$ N-solution of the same salt. The value 0·69 volt has been provisionally adopted as the end-voltage of the titration, this value having been obtained by titrating $\frac{N}{10}$ acetic acid solution with $\frac{N}{10}$ sodium hydroxide solution, with phenolphthaleïn as indicator; and this value also closely corresponds to a hydrion concentration equal to that of pure water. In measuring the difference of potential between the calomel and hydrogen electrodes, the two ends, P and Q, of a sliding rheostat are attached to the terminals of a dry cell, D, and the potential difference to be measured is balanced against the potential difference between one end, P, and the slider, S, by means of the capillary electrometer, E, the value being then read upon a sensitive voltmeter, V. The terminals, X and Y, are connected with a dry cell, and the terminals, Z and U, with the hydrogen and calomel electrodes. Hydrogen is first passed through the hydrogen electrode until the potential difference between it and the calomel electrode becomes constant. This potential difference is now measured by moving the slider up and down until, on depressing the key, K, no movement of mercury in the capillary electrometer is seen. The initial P.D. is then read on the voltmeter scale, which ranges from 0 to 1·5 volt. Alkali is now run in from a burette until, after establishment of equilibrium, a reading of 0·69 is shown on the voltmeter, it being then assumed that the liquid has been neutralised. The initial reading of the tan-liquors examined was usually about 0·54 volt. The following test estimations, in which known quantities of $\frac{N}{10}$ acetic, lactic, or butyric acid were added to a tan-liquor which had previously been titrated electrometrically, illustrate the accuracy of the method:

Acetic Acid.		Lactic Acid.		Butyric Acid.	
C.C. added.	C.C. found.	C.C. added.	C.C. found.	C.C. added.	C.C. found.
5	4·9	5	5·0	9·9	9·85
10	9·9, 10·0, 10·05	10	9·90, 9·95, 9·90	14·85	14·75
15	15·1	15	15·08, 15·05	—	—

Comparative estimations of the acidity of various samples by the electrometric method and by Procter's lime-water method gave the following results:

C.C. $\frac{N}{10}$ ALKALI SOLUTION REQUIRED TO NEUTRALISE 10 C.C. OF THE LIQUOR.

Electrometric method	7·0	9·1	10·0	10·6	10·3	10·45	10·65
Lime-water ,,	5·88	8·24	8·75	9·4	9·3	9·40	9·26

C. A. M.

Quantitative Estimation of Tannin by Means of Casein. M. Nierenstein. (*Chem. Zeit.*, 1911, **35**, 31.)—Experiments upon the estimation of tannin in barks and fruits by absorption with casein gave results from 1 to 1·5 per cent. higher than those obtained by the hide-powder method. The presence of alumina in decolorised tannin extracts caused the non-tannin substances to be too high. On the other hand, absorption by casein may prove of use in the analysis of tannin solutions in the dyeing and textile industries, and experiments are described which show that casein precipitates pure tannin quantitatively from [illegible] solutions, but does not form an insoluble compound with gallic acid or dextrose, both of which are frequently found associated with tannin. C. A. M.

INORGANIC ANALYSIS.

Estimation of Aluminium and Chromium in Ferro-Vanadium. W. Trautmann. (*Stahl u. Eisen*, 1910, **30**, 1802-1803; through *Chem. Zentralb.*, 1910, **2**, 1947-1948.)—The powdered alloy is fused in a nickel crucible, with equal parts of carbonate and peroxide of sodium, the melt is dissolved in hot water, and the diluted solution filtered into a beaker containing hydrochloric acid. Excess of ammonia and a little hydrogen peroxide are added to the acid solution, and the precipitate of alumina, which is contaminated by vanadium, is filtered off. The precipitate is freed from most of the vanadium by washing with an ammoniacal solution of ammonium nitrate containing some hydrogen peroxide. To remove the last traces of vanadium, the precipitate is redissolved in hydrochloric acid, and, after addition of ammonium nitrate and hydrogen peroxide, is reprecipitated by ammonia. The alumina is washed free from chloride by means of a hot ammoniacal solution of ammonium nitrate. Finally, the ignited alumina is freed from co-precipitated silica by means of hydrogen fluoride or by fusing with pyrosulphate.

To separate chromium from vanadium, the filtrate from the peroxide fusion is acidified with sulphuric acid, reduced by sulphur dioxide whilst boiling, and the chromium precipitated by ammonia. On redissolving the precipitate in dilute sulphuric acid, and again precipitating with ammonia, the chromium hydroxide is obtained quite free from vanadium. G. C. J.

Analysis of Bog-Iron Ores. A. Kaysser. (*Chem. Zeit.*, 1911, **35**, 94.)—The analysis of bog-iron ores presents many unsatisfactory features, especially as no confirmatory check can be applied to control the accuracy of the results. The sampling gives no difficulty, since the ore is easily pounded; but, in grinding the sample for analysis, the grains of free silica present tend to work to the top, and the composition of the portion taken for analysis is apt to vary with every movement of the bottle. The grinding must therefore be as fine as possible, and is best performed by a mechanical ore-grinder. The sample should then be dried for at least five hours at 100° C., taking into account the fact that at higher temperatures water of constitution is expelled and organic matter is lost. The dried sample is very hygroscopic, and must be weighed rapidly. In order to cope with the above difficulties, it is recommended that, in performing the assays in duplicate, one portion of 2·5 grms. should be weighed out air-dry, and another portion of 1 grm. after drying at 100° C. The sample should be weighed in a porcelain crucible and then roasted, to destroy organic matter. This should be done in a muffle, first heating the sample carefully, and then placing it in the red-hot furnace; otherwise the ore will decrepitate, on account of the large proportion of moisture present. In order to avoid roasting, the ore may be dissolved in hydrochloric acid, and the organic matter destroyed by a concentrated solution of permanganate. The large quantity of chlorine thus produced may be entirely eliminated by means of the stannous chloride employed for reducing the solution according to Reinhardt's method. In the titration it is desirable to colour the liquid in the basin before titrating with permanganate. A check on the results may be obtained by roasting the 2·5 grms. portion and treating the 1 grm. sample with permanganate, and averaging the results.

J. F. B.

Analysis of Ferro-Zircon. W. Trautmann. (*Zeitsch. angew. Chem.*, 1911, 24, 62.)—For alloys containing less than 20 per cent. of zirconium, the following methods are recommended:

If the alloy is to be opened up by a fusion, it is advisable to roast it first, and then to treat with soda and a little nitre. The alloy oxidises on roasting far more readily than does pure zirconium. On taking up the melt with water, a difficultly soluble compound of zirconium and potassium separates, but may be brought into solution by addition of a few drops of hydrochloric acid. Silica is separated by repeated evaporation with hydrochloric acid, and, when ignited and weighed, is fused with potassium bisulphate, and the melt treated with water and a little hydrochloric acid. The filtrate from the silica, thus purified, contains as a rule from 1 to 3 per cent. of the total zirconium present. For separating the zirconium from the united filtrates, precipitation by means of thiosulphate is preferred. The solution is nearly neutralised, sufficient sodium thiosulphate is added, and when the colour of the solution has been discharged the mixture is heated to boiling, when the whole of the zirconium falls out of solution, together with much sulphur, any aluminium which may be present, and always some iron. The iron is eliminated from the precipitate, after filtration and ignition, by means of a bisulphate fusion, followed by reprecipitation of the zirconium (and any aluminium) by means of thiosulphate. Finally, any aluminium present is separated by means of a peroxide fusion, the aluminium being precipitated from the aqueous extract of the melt by acidifying this with hydrochloric acid, and then adding ammonia. The alumina is weighed, and the zirconium dioxide in the precipitate subjected to fusion with peroxide is determined by difference.

If the alloy is to be decomposed by means of hydrofluoric acid, preliminary roasting is again recommended, especially if the alloy is one containing much carbon. After warming with hydrofluoric and sulphuric acids in a covered crucible until decomposition is complete, the heat is increased, and continued until fumes of sulphuric acid are given off. After the contents of the crucible have been brought into solution in water, with the addition of a few drops of hydrochloric acid if necessary, the zirconium can be estimated in any convenient manner. Duplicate determinations made by the two methods described agree within 0·1 per cent.

For the estimation of carbon in such alloys, direct combustion in a current of oxygen is recommended, the use of lead peroxide being unnecessary. G. C. J.

Analysis of Ferro-Uranium. W. Trautmann. (*Zeitsch. angew. Chem.*, 1911, **24**, 61-62.)—The powdered alloy is treated with hot aqua regia, if necessary for three hours, though a much shorter time suffices for its complete disintegration unless it contains much carbon or silicon. Disregarding any silica which may separate, the solution is diluted and, when cool, solid ammonium carbonate is added in quantity more than sufficient to neutralise the acid present. After allowing to stand for twenty-four hours, with frequent stirring, the precipitate, which contains the whole of the iron, aluminium, and silica, is separated by filtration, and the filtrate boiled for some time to decompose the ammonium carbonate. The greater part of the uranium separates out under these conditions, the solution at the same time acquiring a weak acid reaction. Addition of a few drops of ammonia changes the reaction and determines the quantitative precipitation of the uranium. The precipitate is washed with a weakly ammoniacal solution of ammonium nitrate, transferred to a porcelain crucible, ignited, and weighed as U_3O_8.

Carbon in such alloys is best determined by direct combustion, silicon by repeated evaporation with hydrochloric acid, after opening up with aqua regia. Aluminium, a common impurity, is most conveniently determined after a peroxide fusion. The filtrate from the aqueous extract of the melt is acidified with hydrochloric acid, the alumina precipitated by means of ammonia, and separated from the associated silica by treatment with hydrofluoric acid, or by means of a bisulphate fusion.

G. C. J.

Testing of Incandescent Mantles. J. H. Coste and W. E. F. Powney. (*J. Soc. Chem. Ind.*, 1911, **30**, 65-67.)—In the testing of incandescent mantles, the durability tests precede the photometric tests, since the latter occupy some considerable time, and should only be conducted on those batches which have passed the former test. For the durability test the Woodall-Moon machine is employed. The mantle, having been burnt off and hardened, is subjected on its proper burner to a series of shocks, administered by a miniature stamp-mill operated by a water-motor. The impacts delivered on the lower part of the machine impart to the support and the suspended mantle vibrations similar to those of heavy traffic, and the number is registered by a counter. From each batch of twelve mantles three are tested; and if any fail to survive 1,000 shocks, another three are taken. For upright mantles a minimum of 1,000 shocks, at the rate of 300 per minute, using 2-ounce weights, is exacted; for inverted mantles, 3,000 shocks, at 600 per minute, with 3-ounce weights, constitute the minimum. The shocks are in no case continued beyond 1,200 and 3,600 respectively; this allows one mantle out of six to fail if the other five pass the maximum test. For the photometric tests three mantles are taken from each batch, and burnt on the standard burners for 100 hours. They are then tested photometrically against a ten-candle Harcourt pentane lamp, using a Leeson star disc as modified by Dibdin. Readings are taken at equal angular intervals round the burner, the gas being burnt at the rate of 3½ feet per hour. In interpreting the results it is not desirable to take the average values, since the variations from the average may be very large in the case of individual mantles. The mean variation, obtained by the method of mean squares, is therefore deducted from the average in arriving at the comparative figure of merit. J. F. B.

Reagent Paper for the Volumetric Estimation of Zinc. R. Kopenhague. (*Ann. Chim. anal. appl.*, 1911, **16**, 10-12.)—A paper impregnated with cadmium nitrate enables the end-point of the reaction in the titration of zinc with sodium sulphide to be ascertained much more exactly than is possible with the ordinary lead carbonate paper or the cobaltous chloride paper of Deus. The cadmium paper is prepared by immersing strips of filter-paper in 10 per cent. cadmium nitrate solution, and drying them at 80° to 90° C. It keeps well, and in spotting tests reacts with an excess of sodium sulphide in fifteen to twenty seconds.

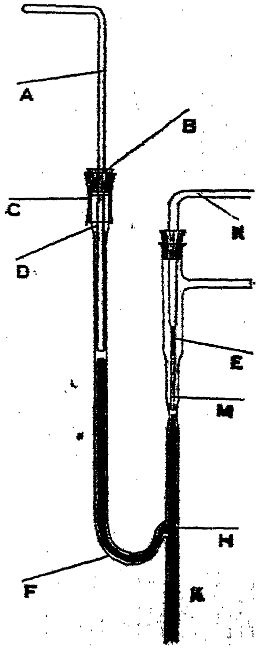
C. A. M.

Estimation of Tungsten. B. Mdivani. (*Bull. Soc. Chim. Belg.*, 1911, **25**, 41-42.)—The tungsten, which may be in solution as tungstate of ammonium or of a fixed alkali, is precipitated as W_2O_5 by addition of a freshly-prepared solution of stannous chloride containing 250 grms. of the crystallised salt in 1,000 c.c. of concentrated hydrochloric acid. About 20 c.c. of this solution are used for each decigram of tungstic acid supposed to be present. After boiling for one or two minutes, the precipitate is allowed to settle, which it does rapidly, and it is possible in a few minutes to wash it several times by decantation with hot water. If the supernatant liquor be decanted on to the filter each time whilst still quite hot, the precipitate remains flocculent, and no difficulty is experienced in filtration. The oxide thus precipitated and washed is finally brought on to the filter, dried, ignited, and weighed as WO_3. The error of the method is ± 0·5 mgrm. on 0·1 grm. WO_3, and the precipitate need never contain a detectable trace of tin. Ferric oxide up to ten times the amount of tungstic acid present does not interfere. G. C. J.

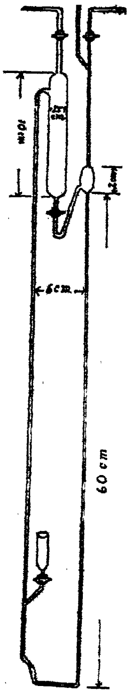
APPARATUS, ETC.

New Form of Constant Temperature Drying Oven. R. L. Siau. (*J. Soc. Chem. Ind.*, 1911, **30**, 61.)—The drying chamber consists of a long, narrow copper tube of round or square cross-section, closed at one end by a plate through which a small tube passes. It is wrapped round by a narrow copper tube of ⅛ to ¼ inch bore which enters the chamber at the end opposite to that closed by the plate and tubulure. A copper jacket of similar section to the oven fits loosely over the coil, the loose end of which passes through the jacket at any convenient point. This jacket is connected below with a boiling flask, and is provided at the top with a Soxhlet's ball condenser. The jacket covers the closed end of the drying chamber, the small tube passing through its wall; whilst at the other end the jacket is sealed off flush with the end of the drying tube by means of a stout plate, forming the entrance to the oven, which can be closed with a clamped door. When in use, the small tube at the farther end of the drying chamber is connected with a water-pump, and air is thereby aspirated through the copper coil, which is surrounded by the vapour in the jacketed space. This air enters at the front end of the oven, and passes over the substances contained therein. The air should be previously dried by suitable drying media, and the moisture expelled from the substance may be determined at the exit end by absorption. The dimensions of the oven are 18 inches in length, 3 inches in width, and 1½ inches in height inside; the boiling flask has a capacity of 250 c.c., and is half filled with water for a temperature of 100° C., or with toluene for 110° C. The moisture in a sample of barley or malt may thus be determined in one and a half to two hours at 100° C., as compared with four to six hours in the ordinary oven. J. F. B.

Gas Regulator for Thermostats. A. Slator. (*J. Soc. Chem. Ind.*, 1911, **30**, 61-62.)—The author describes the following modification of a toluene gas regulator as used on thermostats, which allows the amount of mercury to be decreased or increased according to whether a high or low temperature be desired. It consists of a side-piece, as illustrated in the accompanying figure (in which the toluene bulb is not shown). The glass rod plunger A passes through the rubber bung B, and forces the desired amount of mercury into the stem K of the apparatus. The cup D is filled, to the exclusion of all air, by a mixture of 2 parts of glycerol to 1 part of water. The bung B rests on the cup, and is held in position by the rubber tubing C, which overlaps the bung and cup. The bend F insures the mercury remaining in the side-piece even if the level in the stem falls below the joint H. By means of this arrangement the regulator can be adjusted accurately to any desired temperature over a wide range. It has also been found that the fragile end M of the tube N is better preserved from breakage if the two are connected by means of narrow rubber tubing, E, instead of being rigidly attached. J. F. B.



Freezing Mixtures. J. Duclaux. (*Comptes rend.*, 1910, **151**, 715-716.)—When acetone and carbon bisulphide are mixed, a considerable fall of temperature results. If each of these liquids is conducted separately, at the rate of 1 or 2 drops a second, through a long tube 1 mm. in diameter, to the bottom of a tube closed at its lower end, and wide enough to accommodate the two leading tubes, and if the liquids after mixing are allowed to ascend the outer tube throughout its length, thus cooling the entering currents, a temperature of −48° C. may be obtained. The author makes use of the principle as follows: The two liquids are introduced at the rate of 100 c.c. carbon bisulphide and 70 c.c. acetone per hour into the jacket of a jacketed vessel of silvered glass, the capacity of which, apart from the jacket, is 20 c.c. Once regulated, the temperature of the apparatus and of any substance placed in the vessel can be maintained for several hours at any temperature down to −43·5° without attention. The two liquids separate almost completely on adding to the mixture half its own volume of water. G. C. J.



Modified Boltwood Vacuum Pump. A. F. Odell.

(*J. Amer. Chem. Soc.*, 1911, **33**, 56.)—This modification of Boltwood's pump (*Amer. Chem. J.*, 1897, 76) occupies only one-third the space, and requires only one-tenth the amount of mercury, of the original form, and is equally effective. The pump shown in the figure requires 100 grms. of mercury, and within fifteen minutes a high vacuum can be obtained.

Considerable deviation exists in the manufactured forms of the Boltwood pump from the prescribed internal diameter (2·5 mm.) of the dropping-tube. According to the author, a slightly larger tube refuses to give low vacua, while a tube smaller in diameter than 2·5 mm. works so slowly as to be comparable with the older hand-lift varieties. A. R. T.

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GOVERNMENT REPORTS.

On the Work of Inspectors of Foods. G. S. Buchanan.

(Appendix A, No. 10, from the Annual Report of the Medical Officer of the Local Government Board for 1909-10.)—The Report now submitted deals with work in special relation to the Public Health (Regulations as to Food) Act, 1907, local arrangements for meat and food inspection, work in special relation to the Sale of Food and Drugs Acts, and with foreign and colonial food legislation.

Since the issue of the report on the application of formaldehyde to meat (Analyst, 1909, **34**, 470), the quantity of formaldehyde in certain consignments of imported meat has shown a marked decrease; but there is still room for improvement, if this process is to continue to be applied.

Several inquiries were made as regards the local administration of the Sale of

Food and Drugs Acts. In one instance the attention of the County Council was directed to the undesirability of taking on one day the whole of the samples submitted for analysis during the quarter. In certain counties and boroughs the question of the selection of samples for analysis called for consideration. An inquiry was made regarding a process for reconstructing milk after separation and concentration; this process is carried out on a commercial scale at an establishment near London, and the concentrated separated milk and cream are despatched to London, where they are mixed with any desired proportion of boiled tap-water, and distributed in bulk or in bottles.* It is considered desirable that such reconstructed milk should be sold as prepared milk. The process does not destroy pathogenic organisms which may have been present in the original milk, neither is the reconstructed milk exceptionally free from bacteria.

Importers, rice millers, and other traders, have taken steps to minimise the quantity of steatite employed for "facing" rice since the issue of a Report dealing with the practice (ANALYST, 1909, **34**, 430). The question, however, appears to deserve the continued attention of local authorities, and it may be hoped that their action will restrain the extension of "facing" to other cereals, such as pearl barley.

During the year several public analysts reported on the presence of excessive quantities of tin in samples of canned fruits; many of the cans were badly soldered, and the tops and bottoms were put on so as to leave a raw edge of the metal plate exposed to the fruit-juices. It is desirable that all exposure of solder in the interior of the can should be avoided, and that the inside of the can should be lacquered. Fruits are often packed in glass, but it cannot always be assumed that this has been the original packing. An investigation of a series of bottled fruits for the detection of metallic impurity might be undertaken with advantage in districts where bottled fruit is largely sold.

The question of the issue of regulations regarding the practice of preserving cream by the addition of boric acid or other boron compounds is under the Board's consideration.

Inquiries are now in progress in regard to condensed milk, the composition of food for infants, and the chemical treatment of flour. The experimental work necessary in these inquiries has been facilitated by the provision of a chemical laboratory at the Board's offices. W. P. S.

On a Parasite Condition (Onchocerciasis) met with in Australian Beef. A. W. J. MacFadden and R. T. Leiper. (Local Government Board Food Reports, 1911, No. 11, 1-16, with three plates.)—Certain nodular masses observed in frozen quarters of meat arriving in this country from Australia were found to be due to the presence of long thread-like worms of the genus *Onchocerca.* The diseased condition is not peculiar to Australian cattle, but occurs also in other animals and in other parts of the world, including the United States of America. The parasites belong to a group of worms that require to be taken up by a biting insect, and to undergo a certain degree of development therein before they can be transmitted to another warm-blooded animal. They do not appear to be capable of surviving the death of the cattle for more than a few hours, and no evidence of vitality of the worm or its embryo has been met with in the case of Australian beef reaching this country. It follows that the direct development of the parasite in man, as a result of eating the affected meat, is impossible. The nodules are the product of changes taking place in the tissues as a result of some acrid toxin excreted by the worms, and the opinion is expressed that, for this reason, their presence in meat intended for human consumption is undesirable.

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* This practice is now discontinued.

W. P. S.

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REVIEW.

A TEXTBOOK OF BOTANY AND PHARMACOGNOSY. By HENRY KRAEMER, Ph.B., Ph.D. Fourth revised and enlarged edition, 1910. Philadelphia and London: J. B. Lippincott and Co. Price 21s. net.

Previous notices of earlier editions of this work have appeared in our pages (*cf.* ANALYST, 1907, **32**, 311; 1909, **34**, 192). In the present edition the botanical part has been carefully revised by Dr. T. Holm. This portion of the work, which is admirably illustrated, is claimed to serve as a course of botany for students of pharmacy and pharmacognosy. The micro-analysis of the important plant constituents is treated in a short section, which contains a number of photographs of microscopic crystals. This work, which is admittedly a standard one, will be most useful to the analyst as a practical guide in the microscopical examination of vegetable drugs and allied products. An important feature of the book is the large number of illustrations—some 3,000 plates and 2,000 figures—which it contains. The plan of the work and the general make-up leave little to be desired.

APRIL, 1911. Vol. XXXVI., No. 4

IARI

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE Annual General Meeting of the Society, adjourned from February 1, was resume on Wednesday evening, March 1, in the Chemical Society's Rooms, Burlington House The President, Mr. E. W. Voelcker, A.R.S.M., F.I.C., occupied the chair.

The minutes of the meeting held on February 1 having been read and confirme the election of Officers and Council for 1911 was proceeded with, Messrs. C. A. H B.Sc., F.I.C., and L. Myddelton Nash, F.I.C., being appointed scrutators.

The scrutators having examined the ballot papers, and having reported t result of their examination,

The PRESIDENT announced the election of Officers and Council as follows:

President.—E. W. Voelcker, A.R.S.M., F.I.C.

Past-Presidents serving on the Council (limited by the Society's Articles Association to eight in number).—M. A. Adams, F.R.C.S., F.I.C.; Edward J. Bevar F.I.C.; Bernard Dyer, D.Sc., F.I.C.; Thomas Fairley, F.I.C.; W. W. Fisher, M.A. F.I.C.; Otto Hehner, F.I.C.; R. R. Tatlock, F.I.C.; J. Augustus Voelcker, M.A. B.Sc., Ph.D., F.I.C.

Vice-Presidents.—Arthur E. Ekins, F.I.C.; Arthur R. Ling, F.I.C.; John E. Stead, F.R.S., F.I.C.

Hon. Treasurer.—H. Droop Richmond, F.I.C.

Hon. Secretaries.—A. Chaston Chapman, F.I.C.; P. A. Ellis Richards, F.I.C.

Other Members of Council.—Horatio Ballantyne, F.I.C.; E. Richards Bolton; R. A. Cripps, F.I.C.; F. W. Harbord, A.R.S.M., F.I.C.; Charles A. Hill, B.Sc., F.I.C.; Edward Hinks, B.Sc., F.I.C.; G. N. Huntly, A.R.C.Sc., F.I.C.; F. R. O'Shaughnessy, F.I.C.; Edward Russell, B.Sc., F.I.C.; S. R. Trotman, M.A., F.I.C.; W. H. Willcox, M.D., B.Sc., F.I.C.; S. A. Woodhead, M.Sc., F.I.C.

The proceedings then terminated.

The ordinary monthly meeting followed, the President occupying the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. J. Clifford, A.R.C.S.I., J. Connah, B.Sc., F.I.C., H. F. Knight, C. H. LaWall, W. R. Mummery, F.I.C., and W. M. Seaber, B.Sc., F.I.C., were read for the second time; and certificates in favour of Messrs. Horace G. Battie, 24, Malvern Road, Beeston Hill, Leeds, senior research chemist to Messrs. B. R. Vickers and Son, Leeds;

eorge Davidson Elsdon, B.Sc. (Birm.), City Analyst's Laboratory, Council House, Birmingham, assistant to Mr. J. F. Liverseege, F.I.C.; Norman Evers, B.Sc. (Lond.), A.I.C., Bedcote House, Stourbridge, assistant to Mr. J. F. Liverseege, F.I.C.; and Arthur William Knapp, B.Sc. (Lond. and Birm.), F.I.C., 275, Birchfield Road, Birmingham, analytical chemist, were read for the first time.

Messrs. R. L. Collett, B.A., A.I.C., W. B. Cowie, F. Evershed, and G. Taylor, were elected members of the Society.

The following papers were read: "The Estimation of Iron by Permanganate in e Presence of Hydrochloric Acid," by Alexander Charles Cumming, D.Sc., and exander Gemmell; "Examination of the Process of Shrewsbury and Knapp for e Estimation of Cocoanut Oil," by Raymond Ross, F.I.C., Joseph Race, F.I.C., d Frank Maudsley, B.Sc., F.I.C.; "Note on Henry C. Frey's Method of Estimating etroleum in turpentine," by Herbert S. Shrewsbury, F.I.C.; "Notes on the Formaion of Hypoiodites and their Action on Sodium Thiosulphate—A Source of Error n Certain Iodine Titrations," by J. P. Batey; and "The Analysis of Sweetened ondensed Milk," by Arnold Backe, Ph.D.

A new form of specific gravity apparatus was shown by Dr. C. Butler Savory.

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OTES ON THE FORMATION OF HYPOIODITES AND THEIR ACTION ON SODIUM THIOSULPHATE—A SOURCE OF ERROR IN CERTAIN IODINE TITRATIONS.

By J. P. BATEY, M.Sc.Tech.

(*Read at the Meeting, March* 1, 1911.)

In the course of some analyses of antimonious compounds by titration with iodine in presence of sodium bicarbonate, it was desired to make an indirect titration by adding an excess of the standard iodine solution to the mixture of the antimony and bicarbonate solutions, and then titrating back the excess of iodine by means of a standard solution of sodium thiosulphate. It was found that the results obtained by the indirect titration did not agree exactly with those obtained by direct titration with iodine.

As the indirect titration for antimony affords a much sharper end-reaction than the direct, and is more rapidly carried out, it was thought advisable to ascertain the cause of the observed discrepancy. This was all the more desirable because in a recent paper on the estimation of antimony by Beckett (*Chem. News*, 1910, **102**, 101; Analyst, 1910, **35**, 451) it is stated that both methods of carrying out the estimation yield the same results.

A number of experiments were therefore made, and these, while settling the original point in question, have led to some observations, which may be of interest, on the formation of salts of hypoiodous acid and their oxidising action on sodium thiosulphate.

1. *The Titration of Antimonious Salts with Iodine.*—In one experiment, 25 c of a solution of tartar emetic mixed with 25 c.c. of a saturated solution of sodiu bicarbonate required:

By direct titration ... ... 24·6 c.c. $\frac{N}{10}$ iodine solution.
By indirect titration ... ... 24·85 ,, ,,

In a second experiment, in which the amount of tartar emetic taken was the same, but in which 50 c.c. of the bicarbonate solution was used, the following results were obtained—

By direct titration ... ... 24·6 c.c. iodine solution.
By indirect titration ... . 25·5 ,, ,,

To make quite sure that the observed results were not due to the presence of norn sodium carbonate in the bicarbonate, the saturated solution of the latter was su jected to a current of carbon dioxide for some time. The resulting solution was use but it was still found that the amount of iodine used up in the indirect titration w excessive, although the error was less than before.

It was finally found that the double titration gave quantitative results if t amount of sodium bicarbonate used was reduced to a certain limit. Thus, in a la number of titrations, it has been found that if 1 to 1·5 grms. of bicarbonate (or 20 of a saturated solution) be employed for every 0·15 grm. of antimony present (25 c of an $\frac{N}{10}$ solution), exact results are obtained by the indirect titration; but if mo bicarbonate than this be used, an error is introduced.

It has further been found that Rochelle salt may be conveniently substituted for the bicarbonate in the indirect titration, and in this case the quantity used (provided it be sufficient) is of no consequence, since no error is introduced by using a large excess.

2. *The Titration of Iodine in Presence of Sodium Bicarbonate.*—The following experiments were carried out with the object of determining the cause of the error mentioned above: Standard iodine solution was mixed with sodium bicarbonate, and the iodine then titrated with sodium thiosulphate. A considerable loss of iodine took place, the extent of which is shown in the following table. Decinormal solutions of iodine and sodium thiosulphate were used, and in each case the bicarbonate was weighed out and shaken with sufficient cold water to dissolve it (about 15 c.c. to 1 grm.):

Weight of $NaHCO_3$.	Iodine Solution taken.	Thiosulphate Solution required.	Iodine lost per 1 Grm. of $NaHCO_3$.
grms.	c.c.	c.c.	c.c.
0·0	10	10·05	—
0·5	,,	9·3	1·4
1·0	,,	8·9	1·1
2·0	,,	8·5	0·75
4·0	,,	7·7	0·675

The loss of iodine under these conditions is considerable. A similar effect was observed by Puckner (*Chem. Centr.*, 1905, **1**, 1186).

I found that, if borax were used instead of sodium bicarbonate, a little iodine was absorbed, but not nearly to the same extent as with the bicarbonate. With Rochelle salt, on the other hand, no absorption of iodine could be detected.

It was thought that the loss of iodine might be due to the formation of iodates, and it was expected that, on acidifying the solutions which had been titrated with thiosulphate, the remainder of the iodine would be liberated. Excess of hydrochloric acid was therefore added to each of the solutions after titration with thiosulphate, but in no case was there the slightest trace of iodine liberated. If, therefore, an ate had been formed, the only possible explanation of its disappearance would be at it was reacting with sodium thiosulphate or with sodium tetrathionate. A few periments soon proved, however, that potassium iodate, either alone or in admixre with sodium bicarbonate and potassium iodide, had not the slightest oxidising tion either on sodium thiosulphate or on sodium tetrathionate.

The possibility of the formation of a hypoiodite was next considered, and it was ecided to study the action of sodium hypoiodite on sodium thiosulphate.

3. *The Action of Sodium Hypoiodite on Sodium Thiosulphate.*—R. L. Taylor J. *Chem. Soc.*, 1900, **77**, 725) has shown that, when excess of caustic soda solution is dded to a solution of iodine, the first products of the reaction are sodium hypoiodite nd sodium iodide. The hypoiodite is rapidly resolved into a mixture of iodate and dide. In such a solution an excess of a mineral acid will of course liberate a quanty of iodine equal to that originally taken.

The standard iodine solution was decinormal, and the solution of sodium hydroxide was of normal strength; about 15 c.c. of it were used for every 25 c.c. of iodine.

In studying the action of sodium hypoiodite on sodium thiosulphate, a measured volume of standard iodine was taken, made strongly alkaline with sodium hydroxide, and the desired amount of sodium thiosulphate at once run into the solution. The mixture was shaken and then acidified with hydrochloric acid, and any excess of iodine was titrated by means of sodium thiosulphate. It was at once apparent that when sodium thiosulphate reacts with sodium hypoiodite it uses up far more iodine than would be required to produce sodium tetrathionate.

In one experiment 30 c.c. of iodine was treated with sodium hydroxide, and 5 c.c. of thiosulphate then added. On acidifying, the liberated iodine required only 3·8 c.c. of the $\frac{N}{10}$ thiosulphate, so that in this case 26·2 c.c. of iodine had been used up by 5 c.c. of thiosulphate of the same equivalent strength. When the mixture of sodium hypoiodite and sodium thiosulphate was allowed to stand for a short time before acidifying, the oxidation proceeded still farther, provided of course that a sufficient excess of iodine was used. The limit of the reaction was reached when 1 c.c. of thiosulphate had used up 8 c.c. of iodine. It was found that sulphuric acid was produced, and the equation for the reaction may be represented as follows:

$$Na_2S_2O_3 + 4NaOI + 2NaOH = 2Na_2SO_4 + 4NaI + H_2O.^*$$

4NaOI is equivalent to $4I_2$.

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* This equation of course only represents the ultimate effect of the oxidation. It is probable that several intermediate stages are passed through in the conversion of thiosulphate into sulphate.

In estimating the sulphuric acid produced, 5 c.c. of the $\frac{N}{10}$ sodium thiosulphate (equivalent to 0·233 grm. $BaSO_4$) was run into an excess of hypoiodite solution. After a short time the mixture was acidified with hydrochloric acid, and the sulphuric acid then estimated by the usual gravimetric method. The operation of precipitating and filtering off the barium sulphate was done in the cold. Several estimations of sulphate have been made with solutions which have been allowed to stand for different lengths of time before acidifying. From the amount of sulphuric acid produced in each case the amount of iodine required for the oxidation has been calculated (assuming that that portion of the thiosulphate which is not oxidised to sulphuric acid has been converted into tetrathionate on acidifying). The values thus calculated agree very closely with the loss of iodine actually found by titration.

Iodine used up, found by Titration.	Iodine required, calculated from the Amount of Sulphuric Acid produced.
c.c.	c.c.
22·5	22·30
28·8	28·40
33·6	33·40
34·9	35·30
39·0	38·95

There was still the possibility that potassium iodate in strongly alkaline solution might be capable of bringing about this oxidation of thiosulphate. The following experiments, however, prove that this is not the case, and they afford still further proof of the powerful oxidising action of the sodium hypoiodite.

Twenty c.c. of $\frac{N}{10}$ potassium iodate was mixed with an excess of potassium iodide solution and 20 c.c. of $\frac{N}{1}$ sodium hydroxide. To the mixture 3 c.c. of $\frac{N}{10}$ sodium thiosulphate was added; the solution was then acidified with hydrochloric acid, and the liberated iodine titrated. If no oxidation of thiosulphate had taken place, it was expected that the liberated iodine would require 17 c.c. $(20 - 3)$, but it was found, however, to require only 11·4 c.c. It was at once thought that the potassium iodate was exerting an oxidising action on the thiosulphate, and the experiment was repeated, the mixture being allowed to stand for some time before acidifying. It was expected that still less iodine would be obtained, but instead the titration required 13·9 c.c. of thiosulphate (*i.e.*, more than before).

These curious results were due to the fact that, when the strongly alkaline solution was being acidified with hydrochloric acid, iodine was liberated, which, reacting with local excess of caustic alkali, produced a momentary formation of hypoiodite. It is the hypoiodite alone which brings about the observed oxidation of the thiosulphate. This was shown to be the case by repeating the experiments, and running the alkaline solution from a fine jet, slowly, into a large excess of hydrochloric acid. In several titrations the iodine recovered varied from 16·0 to 16·4 c.c., thus showing that much less oxidation was taking place than before. By preventing all possibility of the formation of hypoiodite, it was possible to recover the total

17 c.c. of iodine, and this was done by first saturating the alkaline mixture with carbon dioxide, when all the alkali present would be converted into bicarbonate, and the solution at the same time saturated with free carbon dioxide. Directly this solution comes in contact with hydrochloric acid, so much carbon dioxide is liberated as to render the momentary formation of hypoiodates impossible.*

The oxidising action of sodium hypoiodite upon sodium tetrathionate† has also been studied, and for this purpose a standard solution of tetrathionate was made up of $\frac{N}{20}$ strength, by mixing equivalent quantities of $\frac{N}{10}$ iodine and $\frac{N}{10}$ thiosulphate.

As in the case of thiosulphate, the oxidation, on standing, was found to proceed to sulphuric acid, so that in this case one equivalent of the tetrathionate (2 c.c.) reacts with seven equivalents of iodine (7 c.c.). The reaction, however, is much slower with tetrathionate than with thiosulphate.

Taking 20 c.c. of iodine in the form of hypoiodite, and adding 6 c.c. of tetrathionate, it was possible, by acidifying the mixture quickly, to recover 19·6 c.c. of the iodine—*i.e.*, only 0·4 c.c. had been used up.

Working under similar conditions with an equivalent amount of thiosulphate (3 c.c.), the least amount of iodine which has so far been used up in any of a large number of experiments was 11·2 c.c. Even if we subtract from 11·2 the 3 c.c. which would be normally required to form tetrathionate, it will still be evident that the reaction between hypoiodite and thiosulphate proceeds under these conditions at something like twenty times the speed of that between hypoiodite and tetrathionate.

From the results of these experiments it would seem very improbable that, in the oxidation of thiosulphate by hypoiodite, the thiosulphate passes through the tetrathionate stage.

4. *The Action of Sodium Bicarbonate on Iodine.*—Returning now to the action of sodium bicarbonate on iodine, it would appear that some sodium hypoiodite is produced. From the researches of R. L. Taylor and others, however, it is evident that the quantity of hypoiodite formed will be diminished by the presence of free carbonic acid. This has been verified experimentally by taking a known weight of sodium bicarbonate and adding to it a known volume of $\frac{N}{1}$ sulphuric acid, thereby liberating some carbon dioxide, then adding iodine and titrating with thiosulphate.

The results are given in the following table:

Weight of Bicarbonate.	Weight of H_2SO_4.	Iodine.	Thiosulphate.
grms.	grm.	c.c.	c.c.
2	0·1	10	9·50
,,	0·2	,,	9·60
,,	0·4	,,	9·80
,,	0·6	,,	9·95

In the above experiments the acid was added very slowly, with the jet of the burette below the surface of the bicarbonate solution.

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* Orton and Blackman (*J. Chem. Soc.*, 1900, 830) have shown that sodium arsensite is oxidised to arsenate by hypoiodites in alkaline solution, but that it is not oxidised by iodates.

† Gutmann (*Ber.*, 1907, **40**, 3614) found that sodium tetrathionate reacted with a hot solution of caustic soda (15 per cent.) to form sodium thiosulphate and sodium sulphite.

In the oxidation of antimonious compounds to antimonic compounds by iodine, ree hydriodic acid is produced, according to the equation:

$$Sb_2O_3 + 2I_2 + 2H_2O = Sb_2O_5 + 4HI.$$

The hydriodic acid reacts with the bicarbonate and liberates carbon dioxide; and if the ratio of free carbon dioxide to sodium bicarbonate is sufficiently great, the formation of hypoiodites is prevented, and therefore the thiosulphate is not oxidised too far. This, therefore, explains the fact observed above, that if the amount of bicarbonate used be reduced to a certain limit the indirect titration for antimony is accurate.

CHEMICAL DEPARTMENT,
SCHOOL OF TECHNOLOGY, MANCHESTER.

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NOTE ON HENRY C. FREY'S METHOD OF ESTIMATING PETROLEUM IN TURPENTINE.

BY HERBERT S. SHREWSBURY, F.I.C.

(Read at the Meeting, March 1, 1911.)

IN Frey's method (*J. Amer. Chem. Soc.*, 1908, **30**, 420), 10 c.c. of the sample are mixed with 30 c.c of aniline, the mixture shaken vigorously for five minutes, and allowed to stand. The petroleum is then measured.

With a preliminary experiment the method appeared so satisfactory that I made the following test experiment with prepared mixtures of turpentine and petroleum:

Per Cent. by Volume of Kerosene.	Per Cent. of Kerosene measured after—		
	Five Minutes.	Thirty Minutes.	Forty Hours.
0	0	0	0
1	0	0	0
5	0	0	0
10	0	0	0
30	30	30	37
50	44	54	55
70	68	70	72
90	80	80	84
95	85	84	84
99	84	84	84

Apparently the method is only applicable when the percentage of kerosene is between 30 and 70, and the result then obtained is probably due to a happy compensation of errors.

Thirty minutes seems to be the maximum time necessary to leave the mixture to settle. The kerosene experimented with was the ordinary pitch oil used in Trinidad for illuminating purposes, with a Zeiss butyro-refractometer figure of 16 at 25° C., and an initial boiling-point of 160° C. The temperature at which the experiments were carried out was 28° C.

DISCUSSION.

Mr. L. M. NASH said that some time previously he tried this method—using, however, ordinary aniline, not anhydrous—but had obtained erratic results. Later on he had repeated his experiments, using anhydrous aniline as recommended by Frey, but had found that this made no difference. He noticed that Mr. Shrewsbury's experiments were made with kerosene, but kerosene was never used for adulterating turpentine. Petroleum spirit was what was used.

Mr. J. H. Coste wrote:

"A method based on the behaviour, when mixed, of three liquids of which only one is a chemical entity, the other two being mixtures of variable composition, does not appear hopeful.

"I think Mr. Shrewsbury has done useful work in showing the unsoundness of this method. It is much to be regretted that such enticingly simple processes based on very slender grounds, either *a priori* or empirical, should be brought forward only to mislead. I find that the method is inapplicable to ordinary turpentine substitutes."

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ANALYSIS OF SWEETENED CONDENSED MILK.

BY ARNOLD BACKE (WITH A. WIBORG AND E. RÖER).

(Read at the Meeting, March 1, 1911.)

ANALYSES of sweetened condensed milk often show results which, even though correct analytical methods have been used, produce a somewhat erroneous impression of the composition of the product. For this reason we deem it useful to call the attention of analysts to certain important facts respecting the analysis of these preparations.

Formerly, when the determination of the fat was performed by extraction with ether after drying, one frequently came across analyses which showed too small a percentage of fat. With the adoption of the Gottlieb method, this difficulty has disappeared, and for the estimation of the other constituents no difficulty exists in arriving at figures sufficiently exact for practical purposes. As regards the determination of the sugar percentage, Kjeldahl's method of calculation must be followed (Report of the Carlsberg Laboratory, iv. 1) to obtain a reliable result.

But if the actual determination of the various constituents does not present any difficulty, other circumstances have to be considered which influence to a considerable extent the result of the analyses of condensed milk as a whole. It is natural that, in a tin of condensed milk which remains undisturbed for a long time, the fat should rise to the surface, and that the rapidity with which it thus rises should depend on the consistency of the milk. This movement being very slow, the separation of the fat produced in this way will not be counteracted in a short time if the tin be inverted. It can further be easily ascertained that a large part of the lactose is not in a state of solution in the condensed milk, but appears in the form of crystals. It frequently happens that condensed milks have a gritty taste, which is due to the presence of crystals of lactose. But even when no gritty sensation is perceived, the lactose is nevertheless separated in microscopic crystals. Whether the condensed milk is gritty or not depends on the size of the crystals. By thinning the milk with cold water and by centrifuging at once, it is possible to separate a large portion of the lactose. If a tin of condensed milk remains undisturbed for a certain time, these crystals sink towards the bottom of the tin. The speed of this downward movement is dependent on the size of the crystals and the consistency of the milk. Generally a little cane-sugar separates, but in much smaller quantities; and since it separates in larger crystals than lactose, they sink to the bottom more rapidly. In accordance with the way in which these various factors—size of the crystals, consistency of the milk, length of time during which the tin has stood—are combined, this separation into zones or layers of different composition in the condensed milk presents itself in various ways. Below follow some examples of milks, all of which are normal, but of different consistencies:

I. Milk Fifteen Days after Manufacture, fairly Thin, not Gritty.

(*a*) Analysis of the whole contents of the tin, well mixed:

Fat	11·40	11·45
Proteins (N × 6·37)	10·20	10·31
Lactose	14·13	14·20
Cane-sugar	37·48	37·52
Mineral matter	2·25	—
Water	24·98	24·90

(*b*) Analysis of the whole contents of the tin previously diluted with water:

Lactose	14·18
Cane-sugar	37·61

(*c*) Analysis by zones:

	1. Thin Layer on the Top of Tin.	2. Main Contents.	3. Thin Layer at Bottom.
Fat	12·09	11·84	11·53
Lactose	13·36	13·90	14·71
Cane-sugar	37·96	37·70	37·53
Water	24·73	24·85	24·85

The figures show that with milk of such a consistency the results obtained are sufficiently exact for practical purposes if the milk contained in the tin is well mixed. There is an appreciable difference between (*c*) and the other analyses. This difference will be alluded to later on.

II. Milk Two and a Half Months after Manufacture, fairly Thin, not Gritty.

(*a*) Analysis of the whole contents of the tin, well mixed:

	I.	II.
Fat	12·20	12·17
Lactose	14·19	14·18
Cane-sugar	35·89	35·95

(*b*) Analyses of zones: These have been chosen differently; the top zone comprises one quarter of the total contents of the tin, the next zone half the total contents, and the bottom zone a quarter:

	Top Zone.	Middle Zone.	Bottom Zone.
Fat	12·68	12·44	11·35
Proteins (N × 6·37)	10·29	10·05	10·03
Lactose	12·23	14·03	16·53
Cane-sugar	36·66	36·01	35·42
Mineral matter	2·14	2·14	2·15
Water	25·66	25·50	24·84

The average figures for the zones being—

Fat	12·23
Lactose	14·21
Cane-sugar	36·02

it can be seen that in this case also fairly exact results can be obtained, provided the milk is well mixed before being analysed.

III. Milk Four and a Half Months after Manufacture, rather Thick.

(*a*) Analysis of the whole contents of the tin, well mixed:

Fat	11·31	11·32
Proteins (N × 6·37)	9·62	—
Lactose	14·35	14·45
Cane-sugar	36·49	36·70
Mineral matter	2·13	—
Water	26·08	—

(*b*) The whole contents of a tin diluted with water:

Lactose	14·13
Cane-sugar	37·25

(*c*) Zones:

Top zone = one quarter of the total contents.
Middle zone = one half " "
Bottom zone = one quarter " "

	Top Zone.	Middle Zone.	Bottom Zone.
Fat	11·58	11·48	11·20
Proteins (N × 6·37)	9·74	9·68	9·59
Lactose	13·59	14·43	15·01
Cane-sugar	37·11	36·93	36·29
Mineral matter	2·15	2·13	2·16
Water	25·47	25·51	25·50

While the thicker consistency of the milk has rendered the separation of the various constituents less pronounced, it is more difficult to obtain a homogeneous sample by mixing.

IV. MILK OF MEDIUM CONSISTENCY, ONE AND A HALF MONTHS AFTER MANUFACTURE, NOT GRITTY.

The sample was divided into five equal zones.

	Top Zone.	Second Zone.	Third Zone.	Fourth Zone.	Fifth Zone (Bottom).
Lactose	13·02	13·45	13·48	13·64	14·54
Cane-sugar	40·20	40·00	40·20	40·07	39·50

V. THIN MILK, SEVEN MONTHS AFTER MANUFACTURE, NOT GRITTY, divided into five zones, the top one of which is one-tenth of the total contents of the tin; the second, third, and fourth, one-sixth; and the fifth, the remainder, or a little less than half the contents of the tin.

	Top Zone.	Second Zone.	Third Zone.	Fourth Zone.	Fifth Zone.
Fat	12·00	10·03	9·49	9·04	8·12
Proteins (Bordas' method)	8·83	8·73	8·54	8·49	8·70
Lactose	6·04	10·43	11·47	12·53	16·43
Cane-sugar	44·23	43·80	43·55	42·82	41·88

From this last sample it is seen what enormous differences may arise with thin milk if the tins remain undisturbed for a long time.

The fall in the percentage of cane-sugar is only apparent, and follows the fall in the percentage of water (not shown in the above table); the cane-sugar is separated only to a very small extent. The excess of lactose, in the later zones depresses the percentage of all the other constituents.

None of the various samples mentioned above were gritty milks. In gritty milks it is found that a deposit forms on the bottom of the tin to an extent depending on the consistency of the milk. If the milk is very thin, a more or less compact layer is formed in a short time at the bottom of the tin. In such a case the only means of obtaining a correct analysis of the milk is to dissolve the whole contents in water.

We have already mentioned that among the lactose crystals there are generally also some cane-sugar crystals. At times this latter sugar amounts to a considerable portion of the separated crystals, but as a rule its percentage is very small.

It is possible that the separation of the constituents which takes place in the tins occurs also during the process of manufacture of the condensed milk, although to a lesser degree, and only under certain conditions. That is why one finds now and again tins of the same manufacture whose contents have not exactly the same composition. This has certainly been the case in our sample No. 1, where *c* does not correspond entirely with the fresh milk from which it has been made.

* * * * *

ERRATA.

Page 98, line 9 from bottom, "*Comp. rend.*, 1814" should read "*Comptes rend.*, 1864."
Page 100, bottom of second column of figures, "0·0500" should read "0·0450."

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Aconitine by Silicotungstic Acid. H. Ribaut. (*Bull. Sci. Pharmac.*, 1910, **17**, 634-639; through *Chem. Zentralbl.*, 1911, **1**, 431.)—On an investigation of the method, the following points were established: The composition of the aconitine silicotungstate depends on that of the liquid in which the precipitate is formed. The latter contains more silicotungstic acid the greater the quantity of nitric or of silicotungstic acid present. These acids also increase the solubility of the aconitine silicotungstate, nitric acid having more effect than silicotungstic acid. Under the conditions prescribed in the French Codex—*i.e.*, in presence of a liquid containing 2·3 per cent. of nitric acid and 0·5 per cent. of free silicotungstic acid—Ecalle's factor, 0·793, gives satisfactory results. For practical purposes, under the prescribed conditions, a correction for the solubility of the precipitate may be dispensed with. The formula of aconitine silicotungstate in no way corresponds with Bertrand's general formula, $SiO_2.12WO_3.2H_2O.4Alkaloid + nH_2O$. The composition given by Ecalle, $SiO_2.12WO_3.2H_2O.3{\cdot}5Aconitine + nH_2O$, only holds good for a definite concentration of the liquid in nitric acid and silicotungstic acid. J. F. B.

Estimation of Atropine. Titration of the Alkaloids in Belladonna Extracts. M. Javillier. (*Bull. Sci. Pharmac.*, 1910, **17**, 629-634; through *Chem. Zentralbl.*, 1911, **1**, 430.)—The alkaloid solution should not be too dilute, since the atropine silicotungstate is not perfectly insoluble in water. The solution is acidified with hydrochloric acid until it contains 1 to 2 per cent. of the acid; a 10 per cent. solution of silicotungstic acid is then added drop by drop, with agitation, avoiding any considerable excess of the reagent. The liquid is allowed to stand for twenty-four hours, the precipitate is filtered or centrifuged off, washed with 1 per cent. hydrochloric acid, and incinerated. The atropine equivalent is found by multiplying the weight by 0·4064, and 0·0048 grm. per 100 c.c. is added to the result, to correct for the solubility of the silicotungstate, disregarding the volume of the washings. The precipitation may be effected either hot or cold, but prolonged boiling must be avoided. This process may be employed for the estimation of atropine in belladonna extracts. In this way the author found that certain commercial extracts showed as much as 55 per cent. less atropine than was found by the volumetric method of the French Pharmacopœia. Examination proved that the silicotungstate method gave the true quantity of atropine present. J. F. B.

Quantitative Estimation of Morphine. R. Gottlieb and O. Steppuhn. (*Arch. exp. Path. u. Pharmak.*, 1910, **64**, 54-66; through *Chem. Zentralbl.*, 1911, **1**, 430.)—The authors describe a method, originally indicated by Rübsamen, for the estimation of morphine. The morphine is extracted from the aqueous solution by means of chloroform, and is titrated according to Gordin's method. The morphine is dissolved in about 200 c.c. of water, and the liquid is made slightly alkaline to phenolphthaleïn; the extraction is then carried out by agitating five times with successive quantities of pure chloroform previously washed with water, 600 c.c. in all being used. If the alkaline reaction be strong, the extraction is less complete and less accurate, in consequence of the formation of acid decomposition products of the chloroform. The chloroform is distilled off from the extract, a measured quantity of $\frac{N}{10}$ sulphuric acid is added to the residue, the latter is dissolved in absolute alcohol, the solution is diluted to 100 c.c. with Wagner's reagent, filtered, and an aliquot portion of the filtrate is taken for titrating back the sulphuric acid in excess. One c.c. of $\frac{N}{10}$ sulphuric acid corresponds to 0·0285 grm. of morphine. Small quantities (0·05 to 0·1 grm.) of morphine may thus be estimated with an accuracy of about 96 per cent.

J. F. B.

Estimation of Nicotine in Concentrated Tobacco Juices. H. Ulex. (*Chem. Zeit.*, 1911, **35**, 121.)—The author describes a method which has been used by him for a number of years. Ten grms. of the sample are weighed out in a porcelain dish, and diluted with 1 to 3 c.c. of water, according to the consistence of the extract. A mixture of 1 part of soda-lime and 5 parts of calcined gypsum is then mixed with the liquid in sufficient quantity to produce a coarse powder. This is ground in a mortar with gentle pressure, and is passed through a hair sieve with about 220 meshes per square cm. The particles remaining on the sieve are again ground with a little more of the mixture, until the whole has been sifted. In mixing the liquid with the soda-lime, the quantity taken should be such that all tendency to heating is avoided. The total mixture will now amount to about 50 grms.; the ammonia will be volatilised, and the free nicotine remain behind. The elimination of the ammonia is completed by exposing the powder over sulphuric acid in a desiccator for about an hour. The nicotine is then distilled over in a current of steam. For the distillation, a tin can of about 3 litres' capacity is employed. This is charged with 1·5 litres of water heated almost to boiling; the dry powder is then introduced quickly, together with 3 to 4 grms. of sodium hydroxide and 4 grms. of paraffin wax to prevent frothing. The can is connected with the condenser, and 1 litre is rapidly distilled off. Distillation is then interrupted, and 1 litre of boiling water is added to the contents of the can, a second litre of distillate being collected in a separate vessel. The nicotine in the distillates is titrated with $\frac{N}{2}$ hydrochloric acid in presence of litmus. The method has been proved to give good results, and no evaporation of nicotine takes place during the elimination of the ammonia in the manner described. If this method appears to yield higher results than some of the methods depending on extraction, this is due to the incompleteness of the extraction, and not to a fault in the method described above (*cf.* ANALYST, 1911, **106**).

J. F. B.

Volumetric Estimation of Quinine in Drugs, etc. J. Katz. (*Ber. Deutsch. Pharm. Ges.*, 1910, **20**, 316-329; through *Chem. Zentralbl.*, 1911, **1**, 44-45.)—The process consists in evaporating an alcoholic solution of the alkaloid with hydrochloric acid, sodium chloride being added in order to expel the excess of the acid, and then titrating the quinine hydrochloride with potassium hydroxide solution, using Poirrier's blue as indicator. As applied to the estimation of quinine in cinchona bark, etc., the process is as follows: Six grms. of the dried and powdered bark are shaken with 15 grms. of chloroform and 5 grms. of a 5 per cent. sodium hydroxide solution for half an hour. Forty-five grms. of ether and about 1 grm. of magnesia are then added, and, after shaking, the liquid portion is passed through a filter. Forty grms. of the filtrate are evaporated to a volume of about 1 c.c., the residue is rinsed into a basin with 9 c.c. of alcohol, 10 drops of hydrochloric acid and 0.25 grm. of sodium chloride are added, and the mixture is evaporated to dryness. A further quantity of alcohol is added, and the evaporation is repeated, the residue being then dissolved in 25 c.c. of alcohol, and the solution titrated with $\frac{N}{10}$ alcoholic potassium hydroxide solution after the addition of 5 drops of a 0·2 per cent. Poirrier's blue solution. The number of c.c. of alkali required for the titration multiplied by 0·405 gives the percentage quantity of quinine in the bark. The addition of the magnesia is for the purpose of precipitating the colouring matters present in the bark.

W. P. S.

Colorimetric Estimation of Strychnine. E. Scandola. (*Boll. Soc. Med.-chirug. di Pavia*, 1910; through *Chem. Zentralbl.*, 1911, 1, 593-594.)—The method depends on the production of a red coloration when strychnine is treated with Mandelin's reagent (1 grm. of ammonium vanadate dissolved in 200 grms. of H_2SO_4,H_2O). The strychnine solution under examination is evaporated to dryness on a water-bath, and the residue is well mixed with 2 c.c. of the reagent. After the red coloration has developed, 10 c.c. of water are added, the solution is transferred to a graduated cylinder, and diluted to a volume of 100 c.c. The coloration is then compared with that produced by a known quantity of strychnine under similar conditions. Substances other than strychnine must not be present. The red coloration remains unaltered for several days; it then changes gradually to violet.

W. P. S.

Analysis and Composition of Jams. F. Hartel and J. Solling. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, 21, 168-196.)—In the analysis of jams estimations of the following constituents afford useful evidence as to the character and quality of the jam: Insoluble matter, soluble solids, total acidity, sugars before and after inversion, sugar-free extractives, starch syrup, mineral matters (total, soluble, and insoluble). The sample should also be tested for the presence of agar, dextrin, added colouring matter, and preservatives. Agar may be detected by boiling 30 grms. of the sample with 270 c.c. of water, filtering the solution, and cooling the filtrate; if agar is present, a precipitate forms within twenty-four hours. The precipitate may be collected, dissolved in a little hot water, and the solution cooled so as to form a jelly. A microscopical examination of the jam should also be made. The starch syrup, when present, was estimated as follows: A solution of the soluble constituents is prepared—100 grms. of the solution containing the soluble solids from 20 grms. of the jam; this solution is employed in the polarimetric determinations from the results of which the quantity of starch syrup present is calculated by means of the formulæ given below. E_D = the soluble solids as calculated from the sp. gr. of the solution; E_J = the soluble solids after inversion, calculated by Clerget's formula (100 grms. of sucrose yielding 105·265 grms. of invert sugar); $[\alpha]_{D20}E_{DV}$ = the specific rotation of E_D before inversion; $[\alpha]_{D20}E_{DN}$ = the specific rotation of E_D after inversion; $[\alpha]_{D20}E_J$ = the specific rotation of E_J; $+134{\cdot}1 = [\alpha]_{D20}$ of water-free starch syrup; $-20{\cdot}42 = [\alpha]_{D20}$ of invert sugar; $St.E_J$ = water-free starch syrup in E_J; St.M = water-free starch syrup in the jam; and w.St.M = the percentage quantity of ordinary starch syrup (containing 18 per cent. of water) in the jam.

$$E = \frac{[([\alpha]_{D20}E_{DV} - [\alpha]_{D20}E_{DN}) \times 1{\cdot}1365 \times 1{\cdot}05265] - [([\alpha]_{D20}E_{DV} - [\alpha]_{D20}E_{DN}) \times 1{\cdot}1365].E_D}{100} + E^D$$

$$[\alpha]_{D20}E_J = \frac{E_D \times [\alpha]_{D20}E_{DN}}{E_J},$$

$$St.E_J = \frac{(100.[\alpha]_{D20}E_J) - (100 \times -20{\cdot}42)}{+134{\cdot}1 - (-20{\cdot}42)},$$

$$St.M = \frac{St.E_J \times E_J}{100}$$

$$w.St.M \quad \frac{St.M \times 100}{82}.$$

Results of analyses of 128 samples of various jams are given, the analyses shown in the following table being selected from the figures recorded:

	Insoluble Matters.	Soluble Solids.	Acidity (as Malic Acid).	Invert Sugar.	Sucrose.	Sugar-free Extractives.	Mineral Matters.	Alkalinity of Mineral Matters.	
								Soluble.	Insoluble.
Raspberry ...	7·40	59·12	1·05	—	—	—	0·355	—	—
Raspberry ...	5·14	70·05	1·08	39·46	26·89	3·70	0·372	4·07	0·87
Raspberry (after removal of juice)	21·68	36·32	—	5·42	20·37	10·53	0·747	7·27	1·79
Apricot	1·90	80·66	1·87	52·53	21·38	6·75	0·567	—	—
Apricot (from dried fruit) ...	3·92	28·08	1·54	—	—	—	1·600	13·81	0·62
Strawberry ...	2·37	73·45	1·22	36·60	32·82	4·03	0·410	4·18	0·99
Cherry	7·42	69·66	1·03	43·00	21·25	5·40	0·448	5·10	1·00
Orange	2·13	70·83	0·12	46·09	23·29	1·44	0·171	1·10	1·10
Quince	5·41	57·95	0·68	30·56	15·76	5·63	0·298	2·90	0·30
Quince jelly ...	0·03	73·20	1·06	54·14	12·58	6·48	0·576	—	—
Pumpkin ...	1·28	63·98	0·03	3·26	58·03	2·69	0·336	—	—
Gooseberry ...	2·74	57·98	1·12	46·90	5·24	5·84	0·270	2·00	0·50

The alkalinity is expressed in c.c. of $\frac{N}{1}$ acid per 100 grms. of jam. The above samples represent genuine jams, many of them having been prepared by the authors themselves. Of the whole number of samples examined, five contained agar, thirty-seven added colouring matters, and twenty starch syrup; many of the jams contained apple pulp, whilst one (a mixed jam) contained beetroot and another (also a mixed jam) rhubarb.

W. P. S.

Adulterant of African Sandal-Wood Oil. W. Lenz. (*Ber. Deutsch. Pharm. Ges.*, 1910, **20**, 351-358; through *Chem. Zentralbl.*, 1911, **1**, 23.)—The bark of the karambusi tree (*Warburgia Stuhlmanni Engl.* Order. *Winteranaceæ Canallaceæ*) is found in the Zanzibar markets along with sandal-wood; it yields an oil which is very similar to sandal-wood oil, and it is possible that it may be used to adulterate the latter oil. When distilled with steam, karambusi bark yields 0·6 per cent. of a viscous, yellowish-red oil, having an odour like that of sandal-wood oil. The oil has a sp. gr. of 0·9864 at 20° C., $[\alpha]_D = -41{\cdot}77$, and $[n]_D = 1{\cdot}51269$; it is soluble in its own volume of 90 per cent. alcohol, but is insoluble in 70 per cent. alcohol. The saponification value of the oil is 11·2, and the acetyl value 100·3. The residue remaining after the distillation of the bark yields mannitol, tannin, bitter substances, etc.

W. P. S.

Improvement in the Method of Malt Analysis. J. O'Sullivan. (*J. Fed. Inst. Brew.*, 1911, **17**, 35-48.)—The O'Sullivan method of estimating starch in malt involves the preliminary removal of the constituents other than starch by means of ether, alcohol, and water, after which the starch originally present is calculated from the maltose and dextrin obtained by treatment with diastase. The new method depends on the fact that extraction with water alone is all that is necessary. The author states that the standard method of analysing malts (*J. Fed. Inst. Brew.*, 1906, **12**, 1), while showing total brewers' extract, gives no information as to the composition of the transformation products, and he points out how this may be ascertained, and also how the transforming power (T.P.) of the malt can be expressed from the composition of the products of hydrolysis yielded by the "starch of the malt." By the following process the estimation of starch in malt can be deduced to within ± 2 per cent.:

Cold-Water Extract.—$\frac{N}{10}$ potassium hydroxide, in place of $\frac{N}{10}$ (*cf.* standard method, *loc. cit.*) ammonia is used. The sp. gr. of the solution is taken = (b). The cupric oxide reducing-power is determined gravimetrically on 20 c.c., and the result expressed as maltose per 100 c.c. (d).

Mash Extract.—Fifty grms. of the malt are warmed in the mashing flask to 60° to 61° C., and 360 c.c. water added at 68° C. The mashing is continued for one hour, and during the last ten minutes the temperature is raised to 75° C. The whole is made up to 515 c.c. at 60°, filtered, the sp. gr. (a) and cupric oxide reducing-power of 3 c.c. of the filtrate determined (c). Then $(a-b)$ = the sp. gr. and $(c-d)$ the grms. of maltose per 100 c.c. of the mash extract due to starch products, and $\frac{(a-b)}{4} - 0{\cdot}04$ = grms. of starch products per 100 c.c. (e) (this 0·04 is deducted for the increased amount of protein matter soluble in the hot mash). $\frac{(c-d)\times 100}{e}$ = percentage of maltose in starch products. The dextrin (f) is obtained from the formula $e-(c-d) = f$; and $\left\{f+\frac{c-d}{1{\cdot}055}\right\} \times 10$ = percentage of starch in the malt, where f = dextrin in grms. per 100 c.c. The transforming power (T.P.) of the malt is taken as the percentage of maltose produced from its own starch, and the percentage of starch in a malt so obtained agrees closely with that obtained by the original O'Sullivan method. All the fat is left in the grains in the new method; it is also shown that the cane-sugar in the malt is not inverted either in the cold or hot water extraction.

The following figures illustrate the nature of the results obtained:

Malt.	Grms. of Starch in 10 Grms. of Malt. Improved Method.	Diastatic Activity by— Improved Method. Per Cent. Maltose on Starch Products.	Diastatic Activity by— Improved Method. T.P. $\frac{\text{Grms. Maltose} \times 100}{\text{Grms. Starch.}}$	Lintner's Method. D.P.
1	4·240	84·05	87·9	51
4	3·532	89·33	93·6	40
7	4·629	87·10	91·2	49
11	4·515	79·10	82·0	58
12	4·506	79·10	82·0	44
18	4·384	58·40	60·2	13
19	4·925	78·90	82·3	26
20	4·890	85·68	89·7	31

The above figures indicate:

(1) In some cases the hydrolysis of the starch has gone beyond the so-called "resting stage," and exceeds 84·4 per cent. of maltose on the starch. (2) That there is no relationship between the D.P. Lintner and the maltose produced. (3) That the T.P. number shows the maltose obtained from the starch of the malt under standard conditions, and is an index of how the malt will behave in practice as regards its starch hydrolysis.

The distiller can calculate by this new method how much alcohol a given malt will produce. The cane-sugar in the cold-water extract of the malt must first be estimated by neutralising the alkaline cold-water extract, and digesting 10 c.c. with 0·01 grm. invertase for an hour at 55° C. The increase in the cupric reducing power as dextrose per 100 c.c., divided by 1·0526, gives the cane-sugar in 10 grms. of malt. The yield of alcohol from starch is 57 per cent.; cane-sugar, 53 per cent.; and approximately from the mixed sugars dextrose, levulose, and maltose, 52 per cent. If, therefore, the starch per cent. is multiplied by 0·57, the cane-sugar by 0·53, and the sugars as maltose by 0·52, the results when added give the theoretical yield of alcohol per cent. from the malt. In a malt 6·3 per cent. of alcohol was obtained from the cold-water extract, as compared with 6·4 calculated, and 28·7 per cent. from the fermentation of the unboiled hot mash, as compared with 28·6 calculated.

H. F. E. H.

New Constant in the Analysis of Pepper. C. Arragon. (*Schweiz. Wochenschr. Chem. Pharm.*, 1911, **49**, 46-47; through *Chem. Zentralbl.*, 1911, **1**, 593.)—A determination of the iodine value of a sample of pepper is recommended as affording a means of ascertaining the purity of the sample. If foreign starch be present, the iodine value gives, approximately, the quantity of the adulterant. Two grms. of the finely powdered sample are mixed with 15 c.c. of chloroform and 25 c.c. of Hübl's reagent, and, after four hours' contact, the excess of iodine is titrated with thiosulphate solution. During the titration the mixture must be shaken thoroughly, as the powder occludes unabsorbed iodine. Thirty samples of pure pepper yielded iodine values of from 16·1 to 18, whilst four samples, containing 60 per cent. of rice starch, gave iodine values of 7·5. There is advantage in determining the iodine value on the chloroform extract of the sample. W. P. S.

Estimation of Tartaric Acid. A. Heczko. (*Zeitsch. anal. Chem.*, 1911, **50**, 12-21.)—Though Goldenberg's method (ANALYST, 1908, **33**, 200) is in almost universal use, yet serious differences are occasionally recorded between the results obtained by different chemists, and it is shown that the method always tends to give low results (see also Ordonneau, ANALYST, 1911, 81). These low results, which in the most favourable circumstances are seldom less than 0·4 to 0·5 per cent. below the truth, are in the main due to the fact that potassium bitartrate is not wholly insoluble in alcohol, and to the action of neutral potassium tartrate on freshly precipitated calcium carbonate. The errors due to the former cause may be eliminated by treating the potassium bitartrate used for standardising the caustic alkali exactly as though it were a sample of argol to be analysed, instead of titrating it directly. It is also pointed out that, for material of high lime content, the amount of potassium carbonate specified by Goldenberg is insufficient, and in such cases the total tartaric acid may be seriously underestimated. The use of 15 c.c. instead of 10 c.c. of 66 per cent. potassium carbonate as a matter of routine would avoid this occasional source of error. Finally, it is suggested that some of the differences recorded between different workers' results may be due to mechanical loss during the evaporation to 15 c.c. before addition of acetic acid and alcohol. The author conducts this operation in a 300 c.c. Erlenmeyer flask set with its axis horizontal. G. C. J.

Estimation of Total Tartaric Acid. A. Heczko. (*Zeitsch. anal. Chem.*, 1911, **50**, 73-82.)—The results of an investigation of a process proposed by Moszczenski (*J. Soc. Chem. Ind.*, 1898, **17**, 215) are given. Whilst the method is more rapid than the Goldenberg process, and yields accurate results when applied to pure tartrates, in the case of crude tartrates it gives results which are from 1 to 2 per cent. too high. The details of the method are: Three grms. of the sample containing more than 45 per cent. of tartaric acid, or 6 grms. when the tartaric acid is less than 45 per cent., are boiled for two minutes with 20 c.c. of $\frac{N}{2}$ sulphuric acid; after cooling, the mixture is rinsed into a 200 c.c. flask with 93 per cent. alcohol, well shaken, and diluted with alcohol to the mark. When the precipitate has settled, the liquid portion is filtered, and 100 c.c. of the filtrate are transformed to a 300 c.c. Erlenmeyer flask. Three c.c. of glacial acetic acid and 10 drops of a saturated potassium chloride solution are added, and 16 c.c. of an alcoholic potassium acetate solution (20 grms. of the salt to 100 c.c. of alcohol) are run in drop by drop from a burette, with continual stirring. The mixture is stirred for a further five minutes, allowed to settle for thirty minutes, and the liquid portion is then passed through a filter. The precipitate is washed twice with 40 c.c. of 97 per cent. alcohol, brought on to the filter, and there washed with about 80 c.c. of alcohol. The precipitate is then dissolved in 200 c.c. of hot water, and the boiling solution is titrated with $\frac{N}{5}$ potassium hydroxide solution, using litmus-paper as indicator. The alkali solution is standardised on 1·5 grms. of pure potassium hydrogen tartrate after this has been submitted to the above treatment, but using only one half of the quantities of the reagents mentioned. W. P. S.

Estimation of Tin in Canned Foods. H. Schreiber and W. C. Taber. (*U.S. Department of Agriculture, Bureau of Chemistry, Circular No.* 67, February 8, 1911, pp. 1 to 9.)—The authors have critically examined several methods for determining tin in canned meats, fish, and vegetables, and, whilst agreeing that the method of Schryver (ANALYST, 1909, **34**, 121) by moist combustion with sulphuric acid is accurate, yet, they state, it is open to the objection that not more than 25 grms. of material can be digested in one flask, with a consequent large multiplication of any analytical error. The digestion is further stated to be accompanied by foaming of the material, and to require considerable experience. For these reasons the authors prefer their alkali-fusion method, except in the case of salted fish, etc., containing large amounts of salt, which prevents the ready burning off of the carbon after fusion.

The method is conducted as follows: Obtain a homogeneous sample by passing the material through a meat-grinding machine, and treat 100 grms. of the mixed sample with 10 grms. of magnesium oxide in an 8-ounce wrought-iron crucible. Stir thoroughly into the mixture 50 c.c. of an aqueous solution containing 150 grms. sodium hydroxide and 100 grms. sodium carbonate per litre, add 75 c.c. of alcohol (95 per cent.), and again stir. Evaporate the alcohol carefully on the steam-bath, with frequent stirring to avoid frothing and bumping, removing the crucible occasionally from the source of heat if necessary. After a time the material boils quietly. After about one hour's heating at 100° C., transfer the crucible to a hot-plate covered with a thin sheet of asbestos, and gradually evaporate to dryness, the temperature of the asbestos at first registering 130 to 160° C. Finally raise the temperature and boil down to dryness, this requiring about one and a half to two hours. Place the crucible in a cold muffle, heat gradually, finally burning off the carbon completely (two to three hours). When cold, cover the residue in the crucible with water, and, after standing a few minutes, transfer the contents to a beaker, add 40 c.c. of diluted sulphuric acid (1 : 1) to the latter, and wash out the crucible with water and 10 c.c. of this acid, scraping the sides of the crucible with an iron spatula. To the mixed solutions in the beaker now add 50 c.c. of strong sulphuric acid and 30 c.c. of strong nitric acid. Boil briskly on the hot-plate until fumes of sulphuric anhydride are evolved for ten minutes. Allow the liquid to cool, and when just cold (not solidified) carefully run in under the cover-glass small successive quantities of water until violent action is over. Then add 150 to 200 c.c. water, stir, and wash into a litre Erlenmeyer flask, the total volume of the liquid being 300 to 400 c.c. Next pass a stream of hydrogen sulphide gas for a few seconds into the cool liquid, shake the flask, and add ammonia slowly till the black ferrous sulphide just persists on shaking. Make acid with diluted sulphuric acid (1 : 1), adding 10 c.c. acid in excess. Dilute the liquid to 1 litre with boiling water, and continue a rapid stream of hydrogen sulphide for twenty-five minutes, and then cork the flask and allow to stand overnight. Heat the liquid next day at 100° C. for half an hour, with shaking, and after partial cooling filter the solution, washing the precipitate on the paper six times with a solution containing 50 c.c. of glacial acetic acid and 100 c.c. of saturated ammonium acetate solution per litre. Treat the washed precipitate, returned with the paper to the flask, with 100 c.c. of potassium hydroxide solution (20 per cent.), boiling for a few minutes till the solution is clear. Decant the solution through a double filter-paper, washing till the filtrate is colourless and measures about 200-300 c.c. Add 20 c.c. of strong hydrochloric acid to the filtrate, stir, and continue adding hydrochloric acid in presence of phenolphthaleïn till the liquid is just acid; then add 1 c.c. acid in excess, testing the acidity of the solution with litmus-paper. Stir well and heat at 100° C. for twenty minutes, and again leave overnight.

Test the reaction of the clear supernatant liquid with litmus-paper. If not acid, render acid with strong hydrochloric acid, and then add 1 c.c. in excess. If the liquid after standing overnight is acid and turbid, make alkaline with potash, and then acid with 1 c.c. excess. The liquid must not be alkaline nor too acid, or filtration will be difficult. Stir occasionally during half an hour's heating on the steam-bath. Filter, returning the filtrate to the paper again till perfectly clear. If the liquid is vigorously stirred, and then allowed to rest till the precipitate clots, filtration and washing are usually easy. Wash alternately with water and the ammonium acetate wash-water, till the filtrate is free from chlorides. Dry and ignite the precipitate with the paper in a porcelain crucible, cool, and weigh as stannic oxide.

The method gives good results, as shown by the following analyses in comparison with Schryver's method:

	Schryver's Method. Sn per cent.		Alkali-Fusion Method. Sn per cent.
Mushrooms	0·0385; 0·0366; 0·0355	...	0·0370
Herrings in tomato sauce	0·0682	...	0·0664
Beets	0·0859; 0·0847	...	0·0838
Fish	0·0338; 0·0386	...	0·0332

A. R. T.

Analysis of Oils of Turpentine. II. F. W. Richardson and J. E. F. Whitaker. (*J. Soc. Chem. Ind.*, 1911, **30**, 115-117.)—In reply to the criticisms of Coste (ANALYST, 1910, **35**, 112), the authors point out that Borneo petroleum (which is rich in aromatic hydrocarbons) differs widely from American petroleum, the products of which are principally used for the adulteration of turpentine. They have never yet met with any turpentine substitute or adulterant composed of Borneo petroleum spirit, and their information is that for a very long period no Borneo spirit has been imported into Europe. Samples of American and Borneo "daylight petroleum" gave the following results:

	Borneo.		American.
Sp. gr. at 15·5° C.	0·81364	...	0·80192
Refractive index at 15·5° C. ...	1·4549	...	1·44474
Iodine value (Hübl), sixteen hours	1·65	...	—

These differences were much accentuated in the fractions obtained on distillation:

Fraction at—	Volume per Cent.		Sp. Gr. at 15·5° C.		Refractive Index at 15·5° C.	
	Borneo.	American.	Borneo.	American.	Borneo.	American.
°C.						
1. 126-149	8·0	9·8	0·8090	0·7500	1·4533	1·4180
2. 149-154	21·2	3·6	0·8104	0·7548	1·4541	1·4211
3. 154-161	28·5	3·4	0·8114	0·7580	1·4545	1·4229
4. 161-165·5	13·4	3·0	0·8130	0·7618	1·4546	1·4255
5. 165·5-171	10·6	2·8	0·8134	0·7652	1·4547	1·4286
6. 171-177	8·2	2·4	0·8142	0·7713	1·4547	1·4286
Residue	10·1	75·0	0·8236	—	1·4586	—

The barium sulphate value estimated in the following manner showed that Borneo petroleum undoubtedly contains a large proportion of aromatic hydrocarbons. Five c.c. of the liquid were heated for an hour on a water-bath beneath a reflux condenser, with 8 c.c. of a mixture in equal parts of sulphuric acid (sp. gr. 1·82) and fuming sulphuric acid, the flask being shaken from time to time. The liquid was then cooled, diluted, and poured on to 30 grms. of barium carbonate in a large dish, the mixture made up to 500 c.c., and 100 c.c. (=1 c.c. of the original sample), separated by filtration, boiled to decomposed barium bicarbonate, and the barium estimated as sulphate in the filtrate. The number of mgrms. (less 5 mgrms., the correction for the barium sulphite produced by the reduction of the sulphuric acid to sulphurous acid) gives the barium sulphate coefficient. The following average figures were thus obtained: Benzene (commercial), 1083; Borneo petroleum, 523; American petroleum, 99; American motor spirit, 90; and American turpentine, 98. The barium sulphate value would thus detect benzene if used as an adulterant of turpentine oil; whilst if Borneo spirit were to be used for this purpose, the addition might be detected by this value and by the iodine value. Benzene may be separated from turpentine by distillation with the aid of a Young's still-head. Over 90 per cent. of commercial benzene passes over below 82° C., and the distillates show a uniform sp. gr. of 0·884, and a refractive index of 1·499 at 15·5° C. Morrell's modification of Armstrong's method (ANALYST, 1910, **35**, 218), in which the unpolymerised portions are not separated before distillation, does not give more accurate results than the original process of Armstrong. A mixture of unknown composition analysed by the authors' methods and by the method of Armstrong gave the following results:

Oil Constituents.	Present.	R. and W.'s Methods.	Errors.	Armstrong's Method.
	Per Cent.	Per Cent.		Per Cent.
Turpentine ...	47·6	49·9	+2·3	67·2 (diff.)
Petroleum ...	20·0	19·6	−0·4	32·8
Gasolene	21·65	21·0	−0·65	
Benzene	10·75	9·5	−1·25	

On the other hand, polymerisation with sulphuric acid by Armstrong's method is valuable as a qualitative test for detecting petroleum products in turpentine oil. Thus, a sample of turpentine (sp. gr. at 15·5° C., 0·84292; refractive index at 15·5° C., 1·4630; and iodine value, 257) was found by the authors' distillation process to contain 19·6 per cent. of petroleum products. A sample of genuine turpentine and of this suspected sample examined by Armstrong's method yielded small amounts of unpolymerised liquids with the following characteristics:

	Genuine Turpentine.	Suspected Sample.
Sp. gr. at 15·5° C.	0·8594	0·7994
Refractive index at 15·5° C. ...	1·4760	1·4482

Samples of different kinds of turpentine recently examined have given the following results:

	American.	French.	Russian.	Swedish.
Sp. gr. at 15·5° C.	0·86864 to 0·87224	0·86908 and 0·86805	0·36608	0·8650 and 0·86776
Refractive index at 15·5° C.	1·4695 to 1·4722	1·4705 and 1·4706	1·4710	1·4740 and 1·4772
Iodine value (Hübl), sixteen hours ...	353 to 440	402, 402	276	315 and 255
Barium sulphate factor	90 and 98	103 and 113	139	140
Polarimeter reading $[\alpha]_D$	+78°	−32·3° and −31·5°	+12·6°	+12·75°

C. A. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Modification of Heller's Ring Test for the Detection of Albumin in Urine, etc. F. Michel. (*Chem. Zeit.*, 1911, **35**, 183.)—Heller's ring test for albumin is rendered less liable to error by increasing the sp. gr. of the nitric acid by dissolving in it an inert substance, and especially ammonium nitrate. About 2 c.c. of the reagent thus prepared are placed in a test-tube, and 1 c.c. of the urine, or other solution under examination, added drop by drop with the aid of a pipette. In the case of a large amount of albumin being present, a sharply-defined ring is immediately produced, whilst when only traces are present it appears after a short time. On shaking the tube the two layers slowly mix. In the presence of albumin the upper layer becomes turbid, whereas when ordinary nitric acid is used in the test the turbidity first produced on mixing the two liquids speedily dissolves. On heating the turbid solution with continual shaking it becomes clear; but when the tube is again cooled, the albumin once more separates as a more or less pronounced turbidity. In the test with ordinary nitric acid, the albumin is immediately decomposed in the cold, and the solution cannot again be made turbid. If water is added to the upper turbid layer, there is a still more pronounced turbidity. The albumin deposit may be dissolved somewhat more rapidly by adding a large amount of acetic acid before heating. On then cooling the liquid there is only a relatively small turbidity produced, though it becomes more pronounced on adding a large quantity of water. C. A. M.

Colorimetric Detection of Oxidising Substances in Animal Organs. W. Loele. (*Munch. med. Wochenschr.*, 1910, **57**, 2414-2416; through *Chem. Zentralbl.*, 1911, **1**, 38.)—Certain cells, such as leucocytes, epithelium cells from the mouth, etc., yield coloured compounds when treated with an alkaline phenol solution; as this reaction takes place only in the presence of an oxidising agent, it appears that these cells contain an oxydase together with an amino base. A solution containing formaldehyde and naphthol absorbs oxygen from the atmosphere and becomes coloured first green, then blue, and finally grey-black; the colour is changed to red by acids and is soluble in chloroform. The addition of blood-serum, peptone, meat extract, or bile, accelerates the formation of the colour, whilst boiled meat extract is without influence. It is probable that this action of blood-serum, etc., is due to the presence of an oxidising enzyme. W. P. S.

Detection of Salvarsan, and its Differentiation from Inorganic Arsenic in Forensic Cases. G. O. Gaebel. (*Arch. Pharm.*, 1911, **249**, 49-56.)—Salvarsan (containing about 34 per cent. of arsenic) is now injected in doses of 0·6 to 1 grm. in syphilitic cases. Within half an hour of its introduction into the system, excretion by way of the kidneys begins, but several weeks later arsenic can still be detected in the urine, and even after tests fail to detect it there it can still be found in the body for another month. Salvarsan gives Reinsch's, Marsh's, and Gutzeit's tests for arsenic, and may be detected by the biological test with *Penicillium glaucum.* It may be distinguished from mineral arsenic by Bettendorf's reagent, which gives with it a yellow amorphous precipitate remaining unaltered for days. The precipitate dissolves on heating to form a clear solution, which when boiled gradually darkens, but does not give the typical arsenic reactions with hydrogen sulphide, etc. The readiness with which the organic complex in the molecule can be oxidised may be made the basis of several reactions. Thus, iron chloride gives an intense coloration from green to red, perceptible in a dilution of 1 : 15,000. Gold chloride gives a deep red colour; platinum chloride is slowly reduced in the cold; and Nessler's reagent is instantly reduced. The presence of the aromatic amino group in salvarsan may be detected by an azo reaction. Thus, if the solution is acidified with a few drops of hydrochloric acid, cooled to about 0° C., treated with sodium nitrite solution in slight excess (the excess being subsequently removed by the addition of urea till potassium iodide starch paper no longer turns blue), and then with a saturated solution of α-naphthylamine acidified with hydrochloric acid, there is gradually produced a red to violet-red colour, which becomes more intense on standing. In a solution of 1 : 15,000 the coloration does not appear for some hours in the cold, but its appearance is accelerated by heat. The reaction is characteristic of salvarsan when arsenic is detected in the azo colouring matter. To isolate the latter, the solution is saturated with sodium chloride and, after shaking for some minutes and standing for a few hours, is filtered from the precipitate. The contents of the filter are washed with sodium chloride solution, and dissolved in hot dilute hydrochloric acid, and the solution tested for arsenic by Reinsch's or Gutzeit's test. Atoxyl also forms a red azo colouring matter with α-naphthylamine under these conditions, but the coloration is immediately produced. The two compounds may be distinguished by the fact that β-naphthylamine gives no coloration with diazotised salvarsan, whereas in the case of atoxyl there is produced an immediate onion-red azo colouring matter. To the presence of the amino group must also be ascribed the blue coloration obtained when salvarsan solutions are treated as in the indophenol reaction

Concentrated hydrochloric acid gives a precipitate (salting out) with even very dilute solutions of salvarsan, and a nearly insoluble precipitate is produced with mercuric chloride. For the detection of salvarsan in organic material, the latter is digested for some hours at a gentle heat with several times its volume of 96 per cent. alcohol acidified with a few drops of hydrochloric acid. The liquid is then filtered, evaporated to a syrup at a moderate heat, and gradually treated with absolute alcohol until the precipitate ceases to increase, after which it is filtered and the filtrate freed from alcohol. The residue is taken up with water and filtered, and the united yellow-coloured filtrates are tested as described above. In all cases the author has succeeded in identifying the unaltered salvarsan. Experiments upon the quantitative estimation of the arsenic, in which the organic complex of the compound was destroyed by means of potassium chlorate and hydrochloric acid and the liquid distilled in a current of chlorine in the presence of iron chloride, gave fairly concordant results (*e.g.*, 29 and 29·5 per cent.), but deviating too far from the theoretical quantity (34·2 per cent.). The same result was obtained when a mixture of nitric and sulphuric acids was used to destroy the organic matter. A method in which the slightly acidified solution of salvarsan is titrated with $\frac{N}{10}$ iodine solution, with starch solution as indicator, has given promising results, the end-point of the reaction being quite sharp.

C. A. M.

Composition of the Gases of the Blood in Chloroform Anæsthesia. Estimation of the Chloroform. G. A. Buckmaster and J. A. Gardner. (*J. Physiol.*, 1910, **41**, 246-262; through *Chem. Zentralbl.*, 1911, **1**, 343.)—The dark colour of the blood during chloroform anæsthesia is due to the partial saturation with oxygen of the hæmoglobin, which in cases of profound narcosis is reduced to as low as 60 per cent. of the normal quantity. A chart of the respiration movements shows that this partial saturation does not depend upon a retardation of respiration, but, rather, is due to a combination of the chloroform with the blood-corpuscles, which deprives them of their power of conveying oxygen. For the estimation of chloroform in the gases of the blood, the best method was found to be combustion, in the presence of an excess of oxygen and water, by means of a glowing platinum spiral in a vessel closed with mercury. The reaction is as follows:

$$CHCl_3 + 2Hg + O_2 = CO_2 + 2HgCl + HCl.$$

This requires a diminution in volume equal to that of the volume of chloroform and of the carbon dioxide formed therefrom and absorbed by the water. The mean experimental results confirmed this assumption.

C. A. M.

Changes which Mercury Compounds undergo when in Contact with Organic Matters. W. Brunetti. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 92-94.)—When mercury is detected during the course of a toxicological analysis, the question may arise as to the form in which the metal is present in the animal organs under examination. With the object of ascertaining what changes, if any, take place in the composition of various mercury compounds when these have been for some time in contact with organic substances, the author has carried out a number of experiments, the results of which are given below. The various mercury compounds employed were mixed in quantities of about 0·2 grm. with portions of meat-juice prepared by extracting meat with cold water. After ten days' contact at the ordinary temperatures, the mixtures were acidified with tartaric acid and submitted to steam distillation for three hours. The metallic mercury or mercury compound passing over into the distillate was collected and weighed. This procedure showed that certain mercury compounds are decomposed by organic matter, and yield metallic mercury in the distillate, whilst others, though not decomposed, are themselves volatile. For instance, metallic mercury, mercuric sulphide, and mercuric cyanide are not altered by contact with organic matter, nor do they volatilise with steam; yellow mercuric iodide, although not reduced, is slightly volatile in steam, about 1 per cent. of the quantity present being found as such in the distillate. Red mercuric iodide is also volatile in steam, but it is found in the distillate as yellow iodide; about 18 per cent. of the red iodide is thus volatilised. Mercury ointment also yields mercury in the distillate, but the quantity found depends on the fineness of the particles of the metal in the ointment. The other mercury compounds used in the experiments were all reduced, and yielded distillates containing mercury. These compounds included mercuric peptonate (39·15), mercuric albuminate (32·45), mercuric chloride (30·68), mercurous chloride (19·11), mercurous nitrate (15·26), ammonium mercury chloride (15·01), mercuric acetate (11·34), mercuric oxide (8·73), and mercuric nucleinate (trace). The figures in parentheses indicate the quantities of volatile mercury in each case, calculated in percentages of the total amounts present. (*Cf.* Lecco, ANALYST, 1910, **35**, 259.) W. P. S.

Comparative Studies on the Valuation of Pepsin according to the Directions of Various Pharmacopœia. E. Hercod and T. Maben. (*Schweiz. Wochenschr. Chem. Pharm.*, 1911, **49**, 17-23; through *Chem. Zentralbl.*, 1911, **1**, 590-591.) —The experiments were carried out according to the directions prescribed by different Pharmacopœia. When pepsin is allowed to act on albumin or fibrin in the presence of an acid, and the mixture is analysed from time to time, the results obtained show that the amount of syntonin and proteoses diminishes, whilst the quantity of peptone gradually increases, the rate at which these changes take place depending on the proportion of acid to pepsin. As the quantities of acid and pepsin, the temperature and duration of the digestion, and the preparation of the albumin, vary with each Pharmacopœia, one and the same sample of pepsin may show different digestive powers according to the method by which it has been tested. This is seen from the results given in the following table:

Pharmacopœia.	Acidity of Solution, Per Cent. HCl.	Ratio of Acid to Pepsin.	Temperature of Digestion.	Duration of Digestion (Hours).	Time the Albumin is Boiled (Minutes).	Time the Albumin is Dried (Minutes).	Activity of Pepsin (Pepsin : Albumin).
Belgian	0·25	250 : 100	40° C.	1	10	10	1 : 100
British	0·20	5000 : 100	40·5° C.	6	15	12	1 : 2500
German	0·125	125 : 100	45° C.	1	10	10	1 : 100
Italian	0·09	90 : 100	38° to 40° C.	1 to 2	—	—	1 : 100
Swiss	0·20	200 : 100	40° C.	1 to 2	5	15	1 : 100
American	0·30	3500 : 100	52° C.	2½	15	16	1 : 3000
French	0·25	150 : 100	50° C.	6	—	—	1 : 25 (fibrin)

The authors suggest that the test should be carried out under the following conditions, etc., in order that uniform results may be obtained: Acidity of solution 0·25 per cent. hydrochloric acid, ratio of acid to pepsin 250 : 100, temperature of digestion 52° C., time of digestion 2 hours, time the albumin is boiled 10 minutes, time the albumin is dried 15 minutes, ratio of pepsin to albumin 1 : 2000.

W. P. S.

Analysis of Zygadenus Intermedius ("Death Camas"). F. W. Heyl and L. C. Raiford. (*J. Amer. Chem. Soc.*, 1911, **33**, 206-211.)—The several species of *Zygadenus* are the cause of heavy stock losses, particularly amongst sheep, which have occurred in Montana, Wyoming, and other of the North-Western States of America. The animals appear to suffer most during early spring when the plant is in flower, and after rains. The plant, which contains one or more poisonous principles, has a relatively high food-value as a whole, and the bulb contains a large percentage of sugar. From a chemical examination, Slade (*Amer. J. Pharm.*, 1905, **77**, 262) concluded that the poisonous alkaloids sabadine, sabadinine, and veratralbine were present.

During grinding of the dry plants, it was noticed that the "dust" was extremely irritating (possibly due to veratralbine). The following figures were obtained on analysis of the various parts of the plant:

	Leaf.	Flower.	Bulb.	Root.
Moisture	6·91	6·13	7·50	7·56
Starch (diastase method) ...	none	none	23·53	none
Dextrin	3·26	trace	1·40	0·34
Pentosans	10·81	7·95	4·41	12·04
Crude fibre	16·17	10·58	5·08	21·53
Protein	13·25	19·73	6·19	6·78
Ash	8·12	8·91	4·29	18·41
Light petroleum extract ...	2·04	2·80	0·62	0·89
Total ether extract	5·47	5·21	3·18	3·05
Volatile ether extract	1·33	1·07	1·30	0·80
Alcohol extract	35·12	46·27	39·60	—
Analysis of Alcohol Extract:				
Resin	5·03	—	2·58	5·00
Sucrose	5·60	—	18·44	1·02
Reducing sugar (calculated as invert sugar)	5·89	—	6·69	2·45

By using a modified process, similar to the U.S.P. method for the assay of belladonna leaves, the authors obtained the following amounts of crystalline alkaloidal residues: Leaf, (*a*) 0·41 per cent.; (*b*) 0·28 per cent.; (*c*) 0·26 per cent.; bulb, 0·39 per cent. and 0·24 per cent. The investigation is being continued.

A. R. T.

ORGANIC ANALYSIS.

Quantitative Estimation of Acetaldehyde by Means of Pyrrol, and the Application of this Method to the Estimation of Lactic Acid. W. Sobolewa and J. Zaleski. (*Zeitsch. physiol. Chem.*, 1910, **69**, 441-451; through *Chem. Zentralbl.*, 1911, **1**, 509-510.)—An alcoholic solution of pyrrol slightly acidified with hydrochloric acid gives a red coloration with aldehydes. In presence of less than 0·1 per cent. of aldehyde a white turbidity is formed, which may be developed more conveniently by using an aqueous solution of pyrrol, and heating on the water-bath. Pyrrol which has become somewhat yellow by the action of air during storage is better suited for this reaction than the freshly rectified product. By this means a quantity of 0·2 to 0·3 mgrm. of acetaldehyde or formaldehyde may be detected. The test is less sensitive with the higher aldehydes and those of the aromatic series. Ketones yield the same reaction. The test may be employed for quantitative purposes in the following manner: A series of tubes are charged each with 15 c.c. of a solution containing 5 to 10 mgrms. of pyrrol and 1 to 2 per cent. of free hydrochloric acid, and increasing quantities of the aldehyde solution are added to the tubes from a burette. The tubes are shaken, and after standing for half an hour, the tube showing a turbidity with the smallest quantity of aldehyde is observed. This quantity represents the aldehyde equivalent of the pyrrol solution, which is a constant within the limits of 0·035 and 0·015 per cent. concentration of aldehyde, the aldehyde solution having been brought within these limits before making the test. The constant must be established, for the pyrrol solution used, by means of an aldehyde solution of known strength. This solution is standardised by testing it first against the pyrrol solution, then oxidising a portion of the aldehyde by means of a restricted quantity of standard bichromate solution, distilling off the residual aldehyde and testing the distillate again with the pyrrol solution. The difference between the two tests is then calculated in terms of the quantity of aldehyde equivalent to the quantity of bichromate used. In the estimation of lactic acid by Jerusalem's method the authors have applied their pyrrol test for the estimation of the aldehyde produced. Jerusalem's method always yields results which are considerably too low, but its use is recommended on account of its simplicity. J. F. B.

Carbonylferrocyanides: Their Extraction and Analysis. E. Lecocq. (*Bull. Soc. Chim. Belg.*, 1911, **25**, 72-80.)—Carbonylferrocyanides may be obtained, during the course of the manufacture of potassium ferrocyanide, by evaporating the filtrate from the insoluble potassium calcium ferrocyanide. When the liquid is concentrated to a sp. gr. of about 30° Bé., a precipitate is formed, consisting of calcium sulphate, potassium calcium ferrocyanide, and potassium calcium carbonylferrocyanide. Whilst the latter salt is soluble in water, it is insoluble in solutions containing large quantities of calcium salts, such as the concentrated liquid mentioned. On separating the precipitate and washing it with water, the carbonylferrocyanide goes into solution, and can be separated by concentrating the solution and allowing the salt to crystallise. It has the formula, $CaKFeCO(CN)_5,5H_2O$. The quantity of carbonylferrocyanides in crude materials, etc., may be estimated by thoroughly mixing 100 grms. of the sample with 10 grms. of powdered calcium oxide. After four or five hours' contact, the mixture is washed into a flask with 300 c.c. of water, shaken occasionally for several hours, and filtered. After washing the residue with water, the filtrate and washings are evaporated to a volume of about 100 c.c., and 35 grms. of calcium chloride and 3 grms. of potassium chloride are added to the concentrated liquid. The precipitate which forms is collected on a filter when the solution is cold; the filtrate is rejected and the precipitate is extracted with warm water. The solution of the carbonylferrocyanides thus obtained is concentrated to a volume of 25 c.c. in order to precipitate the last traces of the double ferrocyanide which remain in the solution, and the latter is then treated with 10 c.c. of a 10 per cent. potassium hydroxide solution, and 2 c.c. of a 10 per cent. potassium carbonate solution. The precipitated calcium carbonate is removed by filtration, the filtrate, diluted to 100 c.c., and acidified with 5 c.c. of 20 per cent. sulphuric acid, is titrated with a copper sulphate solution which has been standardised by titration against a quantity of pure potassium calcium carbonylferrocyanide prepared as described above; paper impregnated with an iron salt is used as the indicator in the titrations. The result obtained will be in terms of potassium calcium carbonylferrocyanide, but may be expressed as ferric carbonylferrocyanide or violet (in which state the substance exists in the crude materials), by multiplying the result by 0·706. Ferric carbonylferrocyanide, or violet, may be used as a pigment in inks, paints, etc.

W. P. S.

Critical Investigation of the Analytical Methods for the Estimation of Caoutchouc. O. Korneck. (*Gummi Zeit.*, 1910, **25**, 4-9, 42-46, 78-88; through *Chem. Zentralbl.*, 1911, **1**, 511-513.)—The methods according to which the caoutchouc is estimated by difference should only be employed with the greatest caution. Of the processes depending on direct precipitation, Spence's process (ANALYST, 1907, **32**, 424) is the best. Fendler's alcohol precipitation method is not very well suited for African varieties. Investigations of the methods depending on the preparation of derivatives showed that Budde's bromination method gave low results, particularly with respect to the fraction termed "insoluble caoutchouc substance," consisting of pure caoutchouc and proteins, in which the pure caoutchouc largely resisted bromination. As the result of his studies on the nitrosite method, the author has evolved the following modified procedure: One grm. of technically pure caoutchouc, or the material left after extraction of the resins, is finely ground and swollen in 75 c.c. of benzene in a covered beaker at the ordinary temperature, with frequent stirring. As soon as the maximum degree of liquefaction is reached (varying from a few hours to some days), nitrous fumes, prepared from nitric acid, sp. gr. 1·3 and arsenious acid and dried over phosphoric anhydride until dark green in colour, are passed in, whilst the contents of the beaker are stirred with the gas-leading tube, and any lumps of nitrosite which may form are broken down. After treatment, the liquid is allowed to stand for a short time, and the green solution is decanted off through a Gooch crucible containing asbestos. The nitrosite is well washed with benzene, and is again treated with nitrous fumes in presence of 75 c.c. of benzene. The beaker is then covered, and allowed to stand for twenty-four hours. Next, the nitrosite is well broken up by pressing it against the sides of the vessel with the gas-leading tube, the clear liquid is again decanted off through the crucible, and the kneading is continued with fresh benzene until the washings remain colourless. The nitrosite is collected in the crucible, and washed with absolute ether until it becomes a pulverulent yellow mass. The crucible, nitrosite, empty beaker, and gas tube are then dried to a constant weight at 80° C., and weighed. The nitrosite adhering to the utensils is next dissolved in 50 c.c. of warm acetone, which is poured through the filter, and the whole is washed with acetone until free from nitrosite. The same utensils are dried and weighed again. The weight of nitrosite so found is multiplied by $\frac{136}{289}$ for the calculation of pure caoutchouc. This method gives good results with crude caoutchouc.

The author describes a method for the precipitation of the caoutchouc resins in the form of ozonides. When ozone is passed through a solution of crude rubber in carbon tetrachloride, the ozonides of the resins are precipitated at the surface of the solvent, whilst the caoutchouc ozonide remains in solution. On treatment with a current of steam, the resin ozonides are converted into solid decomposition products, which are easily separated from the other constituents, dried, and weighed. The method is obviously not exact, the results being probably too high, but they agree fairly well with results obtained by other methods. J. F. B.

Estimation of Caoutchouc as Tetrabromide. T. Budde. (*Gummi Zeit.*, 1910, **25**, 269-270; through *Chem. Zentralbl.*, 1911, 1, 513-514.)—In the original description of the volumetric modification of the tetrabromide method, the quantity of $\frac{N}{5}$ silver nitrate (25 c.c.) prescribed is not a sufficient excess when 0·15 to 0·20 grm. of crude rubber is taken for treatment. The author assumed that this error on his part would be self evident, but apparently Korneck (see preceding abstract) has overlooked this point, and the low results recorded by him are in part attributable to this cause. Further, the presence of moisture appears to have an unfavourable influence on the results of the tetrabromide method as compared with those of the nitrosite method, since the concordance between the two is almost exact when dry materials are used in bromination. The author has studied the influence of protein on the results of the tetrabromide method, and finds that 100 grms. of protein may combine with 4·6 grms. of bromine, equivalent to 1·95 grm. of caoutchouc. If the quantity of protein in crude rubber may in certain cases amount to 8 to 10 per cent., the error introduced by its combination with bromine would be only 0·195 per cent. The statement that the "insoluble proteins" in rubber do not combine with bromine in the tetrabromide process must therefore be modified, since the proteins do unite with bromine; but the error thereby introduced is negligible (*cf.* ANALYST, 1910, **35**, 267). J. F. B.

Analysis of Crude Caoutchouc. G. Fendler. (*Gummi Zeit.*, 1910, **25**, 311-316, 351-357; through *Chem. Zentralbl.*, 1911, **1**, 514-515.)—In carrying out the nitrosite method, nitrosites of the proteins are formed which are soluble in benzene but not in light petroleum spirit. The author contends that if the criticisms of Korneck (see above) against the use of petroleum spirit be justified in this connection, the error involved is only small. On the other hand, Korneck states that the preparation of the nitrosite may, if desired, be undertaken without previous removal of the resins soluble in acetone. The error introduced by omission to remove the resins is far more considerable than that caused by the presence of the nitrosites of the proteins. In the tetrabromide process, Budde's volumetric modification is not capable, in its present form, of taking the place of the gravimetric procedure. Further, it is essential in this method also to remove the resins before bromination. According to the author, it is possible in nearly all cases to filter the toluene solution of the rubber through paper before proceeding to brominate. From 100 c.c. of the solution it is possible to obtain 10 c.c. of filtrate in quite a short time; this quantity is then available for bromination free from insoluble proteins and other impurities. The author disagrees with Korneck's view as to the value of Spence's method of direct precipitation, the range of application of which must be very restricted. In his view it should be possible to devise a direct method to be applied to the toluene solution prepared as for the tetrabromide method. The only doubt is whether it would be possible to drive off the toluene quantitatively from the filtered solution. By centrifuging the unfiltered toluene solution, it might be possible to estimate accurately the insoluble impurities.

J. F. B.

Rubber of Mascarenhasia Elastica. (*Bull. Imp. Institute*, 1910, **8**, 346-352).—This new species of *Mascarenhasia*, yielding the rubber known in East Africa as "M'goa" or "Goa" rubber, grows in German and Portuguese East Africa, the East Africa Protectorate, and Pemba Island. The raw, moist rubber has the following composition:

	East Africa Protectorate.	Pemba.	Portuguese East Africa.	
			Ball Rubber.	Smoked Sheet Rubber.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Moisture	10·0	15·8	1·4	8·4
Caoutchouc	69·0	71·7	90·8	74·2
Resin	6·1	7·6	4·5	6·9
Proteins	3·5	3·1	2·4	3·5
Insoluble matter	11·4	1·8	0·9	7·0
Ash, per cent.	2·29	1·09	1·90	1·80
Physical properties... ...	Fairly satisfactory.	Good.	Good.	Good.

The high proportion of insoluble matter in certain of the samples is chiefly due to the presence of vegetable impurities, the result of careless collection.

A. R. T.

Modification of the Furfural Method for the Estimation of Pentosans. K. H. Böddener and B. Tollens. (*J. für Landw.*, 1910, **58**, 232-237; through *Chem. Zentralbl.*, 1911, **1**, 427-428.)—After the distillation with hydrochloric acid, the estimation of the furfural may be considerably accelerated by precipitating the phloroglucide hot instead of in the cold. The reaction then proceeds approximately, according to the equation $C_5H_4O_2 + C_6H_6O_3 = C_{11}H_4O_2 + 3H_2O$. The procedure is as follows: Three hundred c.c. of hydrochloric acid, sp. gr. 1·06, are mixed with the acid furfural distillate in a beaker, and double the calculated quantity of phloroglucin, *plus* 0·15 grm., previously dissolved in hydrochloric acid, sp. gr. 1·06, is added. The total volume is then made up to 400 c.c. by means of the same acid. The mixture is heated to 80° to 85° C., and allowed to stand for one and a half to two hours at the ordinary temperature. The precipitate is collected in a Gooch crucible, washed with 150 c.c. of water, dried for four hours at 95° to 98° C., and weighed in a stoppered bottle. A correction of 1 mgrm. is added to the weight of the phloroglucide to compensate for its solubility, and the weight of furfural is calculated by the factor 0·571. Very good results are obtained with pure furfural; but when methyl furfural, derived from materials containing methyl pentosans, is present, the usual Kröber method of cold precipitation is preferred. J. F. B.

Estimation of Pentosans with the Aid of Fehling's Solution. J. T. Flohil. (*Chem. Weekblad*, 1910, **7**, 1057-1063.)—The author has determined the conditions under which Fehling's solution is reduced by furfural in direct proportion to the quantity present, and he makes use of the data in the estimation of pentoses and pentosans. The details of the method are briefly as follows:

The substance to be analysed is distilled with dilute acid, the distillate made up to 400 c.c., and 50 c.c. withdrawn. This is neutralised, 20 c.c. of Fehling's solution added, the liquid made up to 100 c.c. and boiled for thirty-five minutes under a reflux condenser. The reduced cuprous oxide is then estimated gravimetrically, or the unreduced cupric salt is titrated iodometrically. It is necessary to carry out a blank experiment in which the same quantity of salt is present.

Under these conditions it is found that 1 c.c. of $\frac{N}{10}$ sodium thiosulphate solution corresponds with 0·0063 grm. of copper or with 0·0024 grm. of furfural. The weight of metallic copper multiplied by the factor 0·3775 is stated to give the weight of furfural. Estimations of pentosans in such substances as cocoa are cited both by the phloroyhicide method and the new method, and the agreement is excellent.

NOTE.—This paper is written in a somewhat loose manner. Thus the particular acid used for the decomposition of the pentosan is not stated, nor is the dilution of the acid given. Further, although the author directs that the distillate should be made up to 400 c.c., he does not state what the approximate volume of the distillate should be. It is doubtful whether with some substances the whole of the furfural would have passed over when the distillate measured 400 c.c. The factor given by the author, 0·3775, for calculating the weight of furfural from that of the copper, should, according to his other data, be 0·3809. A. R. L.

Pentosans of Certain Wood Fungi. J. L. Wichers and B. Tollens. (*J. für Landw.*, 1910, **58**, 238-242; through *Chem. Zentralbl.* 1911, **1**, 428.)—The method described by Böddener and Tollens (p. 161) for the precipitation of furfural phloroglucide was used side by side with Kröber's method of cold precipitation for the estimation of the pentosans in various wood fungi. The results with the two methods were fairly concordant, but those afforded by Kröber's method were slightly higher and, in the author's opinion, more reliable. The percentage of pentosans ranged, according to the species, from 1·21 per cent. (for *Fomes polymorpha*) to 6·73 (for *Lenzites flaccida*). The tinder fungus (*Fomes fomentarius*) showed 3·34 per cent. of pentosans, the precipitate containing some methylfurfural derivative. By the hydrolysis of this fungus, dextrose and pentoses—probably also some mannose groups —were detected, but no galactose groups. J. F. B.

Gums from Northern Nigeria. (*Bull. Imp. Institute*, 1910, **8**, 352-365.)—Investigations made show that the gum-trees of Northern Nigeria, in the Bornu and Yola Provinces, are principally *Acacia Senegal*, *A. Seyal*, and *A. Sieberiana*—the species which yield the important commercial gums of the Sudan and Senegal. The following table summarises the results obtained by the examination of gums from different species:

Source of Gum.	Moisture.	Ash.	Matter Insoluble in Water.	Strength as Measured by Viscosity.	Colour of Mucilage.
	Per Cent.	Per Cent.	Per Cent.		
BORNU PROVINCE:					
Acacia Senegal	10·24 to 11·48	2·87 to 3·17	1·20 to 1·94	5·36 to 6·66	Almost colourless to pale brown
Acacia Seyal ...	11·19 to 11·42	2·50 to 2·66	0·84 to 1·41	5·86 to 6·66	Brown
YOLA PROVINCE:					
Acacia Suma ...	13·32 to 13·56	2·00 to 2·36	4·10 to 4·50	14·1 to 16·70	Colourless to brown
Acacia Sieberiana	13·63	2·65	0·76	13·30	Pale coloured
SOKOTO PROVINCE:					
Combretum sp. ...	12·90	2·00	1·20	7·80	Dark brown

The methods of examination employed for these gums are those described in Part II., "Selected Reports Imp. Institute" (Colonial Reports, Miscellaneous, No. 63, Cd. 4,971, pp. 140-142). A. R. T.

Estimation of Hordenine in Germinating Barley. T. Torquati. (*Arch. Farmacol. sperim.*, 1910, **10**, 62-71; through *Chem. Zentralbl.*, 1911, **1**, 166.)—The author describes the following method employed by him in investigations on the formation of hordenine in germinating barley: The material is macerated for twelve hours with a dilute solution of tartaric acid, heated for three hours, and then filtered hot. The dried tartaric acid extract is triturated with freshly-ground gypsum, and extracted with alcohol. The alcohol is evaporated, and the residual extract is dissolved in water; the solution is freed from colouring matters and fat by means of ether, it is then made alkaline with potassium carbonate, and is exhausted by shaking with ether. The hordenine is extracted from the concentrated ethereal solution by means of dilute sulphuric acid, and the solution of the sulphate is again made alkaline and extracted with ether. Pure hordenine is obtained by evaporation of the ether. J. F. B.

Estimation of Hydroquinone. J. Pinnow. (*Zeitsch. anal. Chem.*, 1911, **50**, 154-155.)—The estimation of hydroquinone by means of Fehling's solution, with the aid of tables empirically prepared, as in the case of sugars, is only possible in rare cases. The cupric reducing power of the hydroquinone rises and falls, in a series of comparative experiments, with increasing concentration; the cause of the irregularities apparently lies in chemical-kinetic processes. The oxidation equivalents of hydroquinone and of the potassium salt of hydroquinonesulphonic acid are almost identical: 1 molecule of the substance requires 3 atoms of oxygen. The reducing power of hydroquinone towards Fehling's solution is considerably increased by the presence of sulphites (as in photographic developers). With a sufficient quantity of sulphite, the cupric reducing power of hydroquinone is increased one and a half times, and that of the sulphonate is increased one and a quarter times, with the formation of a combined product of oxidation. On the other hand, the estimation of hydroquinone may be carried out with satisfactory results by extraction for three-quarters of an hour with ether in Partheil's apparatus (*Ber.*, 1901, **34**, 3611), and subsequent drying of the extract in the water-oven. Hydroquinone exists in ethereal solution in double molecules; its dissociation coefficient and the coefficient of distribution for the single molecules, between ether and water at 15° C., have been determined by the author. J. F. B.

Quantitative Estimation of Nitric Acid in Vegetable Products. K. Krog and J. Sebelien. (*Chem. Zeit.*, 1911, **35**, 145-146.)—Estimation of the nitric acid in vegetable products rich in sugar (*e.g.*, beetroot) by Tiemann's modification of Schloesing's method gives inaccurate results, owing to the reducing influence of the carbohydrates. Although cane-sugar might be removed by precipitation as barium sucrate, yet the invert sugar present in old beetroots cannot be eliminated in this way. The author has therefore tested the applicability of Busch's method of precipitating the nitric acid with *nitron* (ANALYST, 1905, **30**, 256) from aqueous and alcoholic extracts of the dried and finely divided substance, the latter being obtained by digesting about 10 grms. for three hours in a shaking apparatus with a mixture of 300 c.c. of 96 per cent. alcohol and 200 c.c. of water, and then diluting the liquid to 500 c.c. An aliquot portion (250 c.c.) was then evaporated to a small volume to expel all alcohol (in which the nitrate precipitate is soluble), and then treated with the reagent. On concentrating the extracts (whether aqueous or alcoholic) a persistent turbidity was produced, and to remove this a treatment with animal charcoal, followed by filtration, was necessary. In parallel experiments the method gave 1·63 per cent. of nitric acid (N_2O_5) in the case of an aqueous extract, and 1·65 per cent. with an alcoholic extract, whilst by the method of Schloesing and Tiemann only 1·38 per cent. was obtained. For products such as dried beetroot the method of extraction with dilute alcohol is preferable to extraction with water, since a purer solution is obtained, and it is not necessary to remove proteins by precipitation with copper sulphate. Occasionally an amorphous greyish-white precipitate (quite distinct in appearance from the crystalline *nitron* nitrate precipitate) is produced on adding the reagent to an aqueous extract; and, although this may be prevented by the addition of sulphuric acid, the other precipitate is then more difficult to separate. On the other hand, such amorphous precipitates are not given by alcoholic extracts. When the amount of nitrate is very small concentration of the volume to 80 c.c., as recommended by Busch, is insufficient, and to obtain complete precipitation it is necessary to reduce the volume to 20 to 30 c.c. before adding the *nitron*, and to keep the mixture in ice-water for one to two hours (*cf.* ANALYST, 1907, **32**, 349).

C. A. M.

Analysis of Fats and Oils. American Chemical Society. (*J. Ind. Eng. Chem.*, 1911, **3**, 50-52.)—The Committee on Uniformity of Methods of Analysis puts forward the following methods tentatively, and invites criticism:

MOISTURE AND VOLATILE MATTER—*Weighing Out.*—The sample is softened by heat, but not melted, and is emulsified (*e.g.*, by means of an egg-beaker). The necessary quantity for the determination is weighed out into a watch-glass or shallow glass dish, the sides of which are not more than 1 cm. high.

Method No. 1.—From 5 to 10 grms. are heated to constant weight at 110° C., the weight being considered constant when successive weighings thirty minutes apart do not differ by more than 0·05 per cent.

Method No. 2.—From 5 to 10 grms. are heated for four hours at 50° C. in a vacuum oven under a pressure not exceeding 30 mm. of mercury.

Routine Method.—The sample is heated in an aluminium beaker placed on an asbestos board over a burner, the temperature not being allowed to exceed 130° C. The beaker is rotated to avoid spluttering, and the operation considered at an end by the discontinuance of rising bubbles of steam or by other signs known to users of the method.

MOISTURE.—Ubbelohde's method is recommended. A mixture of 100 grms. of the sample with 100 c.c. of xylene is distilled slowly, and the first 50 c.c. of the distillate collected in a tube graduated in tenths of a cubic centimetre. The percentage of water is read off directly and corrected for the solubility of water in xylene, as to which the committee has an investigation in hand.

SUSPENDED IMPURITIES.—From 5 to 20 grms. are dried in a beaker over asbestos board (the residue from a moisture and volatile matter determination may be taken), and is dissolved in hot petroleum ether (b.p., 50° to 70° C.) by gentle boiling on the water-bath. The undissolved matter is filtered off in a Gooch or porous crucible, washed with boiling petroleum ether, and dried to constant weight.

FREE FATTY ACIDS.—From 5 to 15 grms. are weighed into a 100 c.c. Erlenmeyer flask, and melted if a solid fat. Hot neutral alcohol (100 c.c.) is added, and the mixture titrated with $\frac{N}{10}$ or $\frac{N}{2}$ caustic alkali, using phenolphthaleïn as indicator.

Acid number to be reported as mg. KOH required to neutralise 1 grm. Where the fat is known to have a mean molecular weight of 282 or thereabouts, that figure is to be used in calculating the percentage of free fatty acids, which is to be based on the sample freed from moisture and volatile matter.

The neutral alcohol required is prepared by boiling 1,000 c.c. of 95 per cent. alcohol with 50 grms. caustic soda for six hours under a reflux condenser, allowing to stand twenty-four hours, and distilling.

TITER (*Melting-Point*).—The fat (75 grms.) is saponified by mixing with 60 c.c. of 30 per cent. caustic soda and 75 c.c. of 95 per cent. alcohol (or 120 c.c. water) in an iron or porcelain dish, and boiling down to dryness over a low flame, with constant stirring to prevent scorching. The soap is dissolved in a litre of boiling water and, if alcohol has been used, boiled for forty minutes to remove it, maintaining the volume of the liquid by occasional additions of water. To the soap solution, freed from alcohol, and contained in a porcelain vessel, 100 c.c. of 30 per cent. sulphuric acid are added, and the mixture boiled until the fatty acids form a transparent layer. The fatty acids are collected in a small beaker, placed on the steam bath until the water has settled, decanted into a dry beaker, filtered with the use of a hot-water funnel, and dried for twenty minutes at 105° C. The dried fatty acids are then cooled to within 15° or 20° C. of the expected titer and transferred to a tube 4″ × 1″ and 1 mm. thick in the wall. This tube is placed in a bottle about 6″ × 3″, which is fitted with a cork to hold the tube rigidly. The thermometer, graduated to 0·1° C., is suspended so that it can be used as a stirrer, and the mass is stirred slowly until the mercury remains stationary for thirty seconds. The thermometer is then allowed to hang quietly, with the bulb in the centre of the mass, and the highest point to which the mercury rises is taken as the titer of the fatty acids. The room temperature should be reported.

The fatty acids are tested for complete saponification by boiling 3 c.c. with 15 c.c. of 95 per cent. alcohol in a test-tube, and adding an equal volume of ammonia (sp. gr., 0·96). No turbidity should appear.

UNSAPONIFIABLE MATTER—*Definition*.—The non-fatty constituents of fats and fatty oils, soluble in petroleum ether (b.p., 35° to 50° C.).

Wet Method.—The fat (3 grms.), free from moisture and volatile matter, is weighed into a 150 c.c. flask, and a 30 per cent. excess of strong, nearly colourless, alcoholic potash is added. The mixture is boiled for an hour under a reflux condenser and then transferred to a 100 c.c. stoppered cylinder, made up to 50 c.c. with cold water, and shaken with 30 c.c. petroleum ether. The petroleum ether layer is syphoned off, and the extraction repeated with five separate portions of petroleum ether. The united petroleum ether extracts are washed three times with 20 c.c. of 50 per cent. alcohol, poured into a tared flask, the greater part of the petroleum ether removed by distillation, and the residue dried on the water-bath, and finally in an oven at 105° C.

Dry Method—The fat (5 grms.) is saponified in a 200 c.c. flask by boiling under a reflux condenser with a 30 per cent. excess of alcoholic soda for thirty to sixty minutes, or until saponification is complete. The solution is transferred to a 4-inch basin and dried on a water-bath, and then in an oven for one to two hours at 120° C.

The dried soap is ground up in an agate mortar with 10 to 15 grms. granular anhydrous sodium carbonate, placed in a 33 × 80 mm. extraction thimble, covered with a plug of cotton wool, and redried for an hour at 110° to 120° C. The thimble is then transferred to a Soxhlet extractor and extracted for about ten hours (fifty or more syphonings) with petroleum ether, access of moisture being excluded by attaching a calcium chloride tube to the top of the condenser. The extract is transferred to a tared beaker, the solvent evaporated on the water-bath, and the residue dried in an oven to constant weight. The residue should be tested for complete solubility in petroleum ether.

METALLIC SOAPS.—*Definition.*—Fatty acid compounds with bases other than alkalies. Insoluble in water.

Method No. 1.—The filtrate and washings from the determination of suspended impurities are evaporated to about 200 c.c., and allowed to stand at least twelve hours in a cool place (18° to 20° C.). The separated insoluble metallic soaps are then filtered off on a Gooch crucible, washed with cold petroleum ether (b.p., 35° to 50° C.), dried, and weighed.

Method No. 2.—The filtrate from the determination of suspended impurities is evaporated, burned, and ignited to constant weight. The weight of the ash from metallic soaps thus obtained is to be reported (1) as such, and, considering the ash to consist entirely of lime, it is calculated to normal soap, using 281 as molecular weight of fatty acid, and reported (2) as lime soap.

Criticisms and suggestions should be addressed to W. D. Richardson, 4215, Prairie Avenue, Chicago. G. C. J.

Interpolation Method of Oil Analysis. J. J. Kessler and G. K. Mathiason.

(*J. Ind. Eng. Chem.*, 1911, **3**, 66-72.)—This paper contains a study of the degree of accuracy obtainable in the method of calculating the percentage composition of a mixture of oils from certain physical constants of the mixture. The results obtained with experimental mixtures are given in tabular form and plotted as curves, and it is shown that certain values such as the viscosity, flash-point, and ignition point (fire-test) and possibly other physical constants do not show an additive relationship, and that considerable errors may result from drawing deductions from interpolation of data thus obtained. This also applies to the Maumené test, and the figures indicate that, as at present applied, the results obtained by that test are incapable of enabling a quantitative estimation of the composition of a mixture to be made, as in the method recommended by Stillman (*Engineering Chemistry*, 1900). In the case of the saponification value, however, the relationship is additive, and holds good even for small proportions of one oil in the presence of another. C. A. M.

Estimation of Lecithin in Oil. W. Fresenius and L. Grünhut.

(*Zeitsch. anal. Chem.*, 1911, **50**, 90-106.)—The following process is recommended for the estimation of lecithin in fixed oils and fats, and is based on the extraction of the lecithin by means of alcohol, the quantity of lecithin being calculated from the amount of alcohol-soluble-phosphoric acid obtained. As lecithin is soluble in both oil and alcohol, the quantity extracted from the oil is proportional to the solubility of lecithin in the two liquids; repeated extraction with fresh quantities of alcohol results in practically the whole of the oil being dissolved by the alcohol, and the authors, therefore, extract the oil twice with successive quantities of cold alcohol, the total quantity of lecithin present being calculated from the amount dissolved by alcohol according to the formula:

$$x = \frac{p_1 a_1 o_1 + p_1 v_2 (o_1 a_2 - a_2 a_1)}{v(p_1 o_1 - p_2 o_2).}$$

Fifty grms. of the oil are weighed into a 200 c.c. graduated cylinder and shaken for twenty minutes with 100 c.c. of absolute alcohol. After standing for one hour, the volumes of the oily layer (o_1) and of the alcoholic layer (a_1) are noted. Seventy-five c.c. (v) of the alcoholic layer are now removed by means of a pipette for the first phosphoric acid estimation (p_1); the pipette is then washed with absolute alcohol, the washing being added to the cylinder, and the contents of the latter are diluted with absolute alcohol to the original total volume. The contents of the cylinder are then shaken as before, the volumes of the two layers (o_2 and a_2) are read off, and a further 75 c.c. of the alcoholic layer are drawn off for the second phosphoric acid estimation (p_2). The two quantities of alcohol are evaporated separately in platinum basins, and the dry residues are fused with a mixture of sodium carbonate and sodium nitrate. The phosphoric acid in the fused masses is then precipitated by means of molybdic acid in the usual manner, and the yellow precipitate is ignited, and weighed. The ignited precipitate has the formula, $P_2O_5.24MoO_3$, and the quantity of phosphoric acid is calculated accordingly. Lecithin contains an average of 9·02 per cent. of phosphoric acid (P_2O_5). Two preparations of lecithin in oil examined, contained 3·11 and 7·80 per cent. of lecithin, respectively. W. P. S.

Determination of Origin of Mineral Oils and their Products. N. Chercheffsky. (*Ann. Chim. anal.*, 1911, **16**, 45-50.)—The author has fractionated numerous samples of American, Russian, Roumanian, Galician, and shale oils, and for the fractions having sp. gr. of 0·75, 0·78, 0·80, and 0·82, has determined the mean boiling-point, refractive index, index of solubility (Riche and Halphen), critical temperature of solution, cloud temperature, and iodine number. By fractionating a sample of unknown origin and examining the fraction in a similar manner and comparing them with his tabulated results, it should be possible to fix the origin of the sample. As it is inconvenient to try to collect fractions of predetermined sp. gr., it is suggested that fractions be collected, each representing a range of boiling-point of 20° from, say, 100° C. to 300° C., and that curves be drawn on squared paper through a series of points, each of which has the mean boiling-point of one fraction for abscissa and its sp. gr. for ordinate. The results of the other tests are plotted on the same paper, and the refractive index, etc., for the fraction of sp. gr. 0·75, etc., may then be seen by inspection. The following tables are given:

I. Boiling-Points °C.

Sp. Gr. at 15°/15° C. of Distillate.	American.	Russian.	Roumanian.	Galician.	Shale Oil.
0·75	141	109	108	113	127
0·78	191	159	153	166	168
0·80	227	182	179	202	198
0·82	266	220	208	242	228

II. Refractive Indices at 15° C.

Sp. Gr. at 15°/15° C. of Distillate.	American.	Russian.	Roumanian.	Galician.	Shale Oil.
0·75	1·4182	1·4143	1·4160	1·4174	1·4219
0·78	1·4345	1·4309	1·4334	1·4356	1·4373
0·80	1·4453	1·4419	1·4458	1·4466	1·4469
0·82	1·4564	1·4533	1·4572	1·4586	1·4568

III. Index of Solubility (Riche and Halphen).

Sp. Gr. at 15°/15° C. of Distillate.	American.	Russian.	Roumanian.	Galician.	Shale Oil.
0·75	70	70	68	68	66
0·78	93	75	73	74	74
0·80	117	85	80	94	93
0·82	154	92	91	126	109

IV. Critical Temperature of Solution °C.

Sp. Gr. at 15°/15° C. of Distillate.	American.	Russian.	Roumanian.	Galician.	Shale Oil.
0·75	20	—	—	—	—
0·78	50	36	—	31	31
0·80	69	48	30	53	43
0·82	87	60	42	73	54

V. Cloud Test °C.

Sp. Gr. at 15°/15° C. of Distillate.	American.	Russian.	Roumanian.	Galician.	Shale Oil.
0·75	61	55	52	49	44
0·78	79	66	53	60	55
0·80	91	72	57	76	63
0·82	105	80	64	90	71

The index of solubility is the number of c.c. of a mixture of equal parts of chloroform and 93 per cent. alcohol which just suffice to dissolve 100 c.c. of the fraction under examination. The critical temperature is determined by heating some of the oil with an equal volume of 96·5 per cent. alcohol in a sealed tube until the mixture is just clear, and noting the temperature at which the first sign of cloudiness appears on slow cooling. The cloud test is made in a similar manner, but in an open tube and with acetic anhydride as solvent. It is said that the iodine number of normal petroleum products varies from 0 to 7, whilst that of some American refined products resulting from "cracking" may be as high as 20 to 25. The iodine number of shale products varies with the density of the oil, that of ordinary burning oil being 65·75, whilst the lighter fractions may have values up to 110. G. C. J.

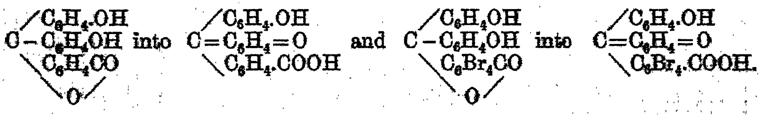
Estimation of Unsaponifiable Matter in Wool Oils. A. H. Gill and A. E. Shippee. (*J. Ind. Eng. Chem.*, 1911, **3**, 72-73.)—The method of extraction with petroleum spirit has the drawbacks that the extraction may be incomplete, that the soap is soluble in the solvent, and that the complete removal of the solvent is difficult, and involves the loss of volatile constituents in the unsaponifiable matter. The other method in common use is based upon an estimation of the free acids in the oil (as oleic acid), followed by an estimation of the combined fatty acids (calculated into olein). The sum of these two values deducted from 100 gives the amount of unsaponifiable matter. The following table gives the results thus obtained by the two methods in the examination of typical samples of wool oils:

Oil.	Sp. Gr. at 15° C.	Unsaponifiable Matter.		Saponification Value.	Free Acids as Oleic Acid.
		Extraction Method.	Differential Method.		
		Per Cent.	Per Cent.		Per Cent.
Scotch wool	0·908	65·7	65·4	73	12·8
Wool oil	0·908	91·7	90·9	17	3·3
Oleine, 42% saponifiable	0·911	58·5	56·6	88	41·3
English oleine, 42% saponifiable ...	0·911	59·4	59·2	80	40·7
Wool oleine, 95% „ ...	0·910	31·2	31·0	140	65·2
Wool oleine, 35% „ ...	0·910	65·2	65·3	69	32·6

Notwithstanding the possible errors due to assuming that the whole of the fatty acids in the oil consist of oleic acid, the results show that for technical purposes the differential method is sufficiently accurate. In fact, with the exception of oil No. 3, the differences between the results of duplicate estimations by either method were greater than the average differences in the results given by the two methods.

C. A. M.

Phenolphthaleïn Derivatives and their Properties as Indicators. E. Rupp. (*Arch. Pharm.*, 1911, **249**, 56-68.)—The halogen derivatives of phenolphthaleïn, such as tetrabromophenol-phthaleïn, in which the halogen enters only into the phenol ring, give only diffused gradual changes of colour, both with ammonia, and with very dilute fixed alkali solutions. On the other hand, derivatives in which the substitution occurs in the phthalic nucleus show a sharp change of colour both with fixed alkalis and ammonia, although this change does not coincide with the saturation-point, but with a minimum of hydroxyl ions which is constant for each particular substitution compound. The explanation of this is given by the chromophore theory of indicators, according to which the formation of coloured salts from colourless acid compounds must be attributed to an intramolecular transformation, the pseudo form of the free phenolphthaleïn being converted into the quinonoid form in the coloured salts—*e.g.*,



$$C\begin{cases}C_6H_4.OH\\ -C_6H_4OH\\ C_6H_4CO\diagdown O\diagup\end{cases}\text{ into }C\begin{cases}C_6H_4.OH\\ =C_6H_4=O\\ C_6H_4.COOH\end{cases}\text{ and }C\begin{cases}C_6H_4OH\\ -C_6H_4OH\\ C_6Br_4CO\diagdown O\diagup\end{cases}\text{ into }C\begin{cases}C_6H_4.OH\\ =C_6H_4=O\\ C_6Br_4.COOH.\end{cases}$$

Since a rapid velocity of isomerisation is essential to the utility of a compound as an indicator, it might be anticipated that octobromophenolphthaleïn would be less suitable for the purpose than tetrabromophenolphthaleïn, and this is borne out by the results of titrations. The fact that phthaleïn derivatives with closed hydroxyl groups, such as diacetylphenolphthaleïn, dibenzoylphenolphthaleïn, and dibenzenesulphophenolphthaleïn, cannot be used as indicators is due to an inhibition of the formation of the colour-producing quinonoid group. Tetrachlorotetrabromophenolphthaleïn gave accurate results as an indicator in the titration of ammonia, quinine, and sodium carbonate, but with this exception only the nitro- and nitrobromo derivatives proved serviceable. In the case of these derivatives, which could also be used with weak bases and with morphine and quinine, the change of colour (yellow instead of red) showed that the property was no longer bound up with the phthaleïn complex, but had passed to the nitro-oxyphenyl group. This was confirmed by a comparative series of titrations of morphine and quinine with *p*-nitrophenol as indicator, the results being in exact agreement with those obtained with these indicators. With regard to their colour relationships, the halogen phthaleïns confirm the general rule that the chromophore bodies pass from red through violet and blue to green with the increase in their molecular weight. Thus, in alkaline solution phenolphthaleïn is red; tetrachlorophenolphthaleïn, red-violet; tetrabromophenolphthaleïn, violet; tetrachlorotetrabromophenolphthaleïn, blue; octobromophenolphthaleïn, greenish-blue; and tetraiodotetrabromophenolphthaleïn, bluish-green. C. A. M.

Colour Reactions of Certain Resins with Halphen's Reagent for Colophony. E. F. Hicks. (*J. Ind. Eng. Chem.*, 1911, **3**, 86-87.)—Halphen's colophony test applied in the manner described by Foerster (ANALYST, 1909, **34**, 108) also gives characteristic colour reactions with certain other resins. The results thus obtained by the author with the pure substances may be summarised as follows:

Colophony.—Green, changing to blue and violet, then slowly to purple, and, finally, to deep indigo.

Dammar.—Brown to lilac-brown, gradually changing to reddish-brown (maroon).

Elemi.—Indigo-blue (immediately), gradually deepening in colour, and sometimes acquiring a purplish tint.

Kauri.—Azure-blue, changing rapidly through violet to purple. At point most remote from the bromine becomes dark olive-green.

Manila Gum (spirit soluble).—Very faint brown-green (slowly), changing through violet to purple. Chocolate-brown at point furthest from bromine.

Mastic.—Reddish-brown, becoming carmine nearest the bromine, and coffee-brown at the most remote point.

Sandarac.—Lilac (immediately). At point remote from bromine gradually changes to violet-brown.

Shellac.—No colorations when pure.

Zanzibar Copal.—Light brown (slowly), changing to brown-violet, and, finally, to chocolate-brown with violet hue.

The intensity of the colours depends upon the proportion of resin present, and the results should be compared with those given by resins of known purity. The test may also give valuable indications in the case of mixtures, though, should colophony be present, the intensity of its reaction will mask the other colorations. The sensitiveness of the test is reduced by the presence of water, alcohol, or ether.

C. A. M.

Lac Resin from Baroda, India. (*Bull. Imp. Institute*, 1910, 8, 371-372.)—The samples were collected from the *Inga Saman*, or "rain-tree" (*Pithecolobium Saman*), which has only recently been used as a host for the lac insect. The following results were obtained on analysis:

	Lac Resin removed from Broken Twigs after Receipt. Per Cent.	Seed-Lac Resin as Received removed from Twigs. Per Cent.
Moisture	2·8	2·7
Ash	1·4	2·2
Wax	9·0	6·6
Colouring matter	7·2	12·4
Insoluble matter	8·1	8·6
Resin	71·5	67·5
IODINE NUMBER:		
Before purification	5·6	4·5
After purifying from dust, etc.	4·0	3·9

(*Cf.* ANALYST, 1911, p. 119.)

A. R. T.

Distillation of Tars Containing Water. C. R. Downs. (*J. Ind. Eng. Chem.*, 1911, 3, 110-111.)—The bumping which results from the falling back of condensed water from the thermometer and neck of the flask when a mixture of tar and water is distilled may be avoided as follows: Two concentric cylinders of 30-mesh brass gauze are made, one just small enough in diameter to fit inside the neck of the flask, the other a little smaller. Both are long enough to extend from the side tube of the distilling flask to a point half-way between the end of the neck and the surface of the liquid, at the commencement of distillation. They are attached by wires to the cork which carries the thermometer.

G. C. J.

INORGANIC ANALYSIS.

Estimation of Arsenic and Antimony in Copper. G. L. Heath. (*J. Ind. Eng. Chem.*, 1911, 3, 78-82.)—The paper includes a criticism of methods which depend on the volatilisation of arsenic (and antimony) chlorides, and describes in great detail the precautions necessary to obtain highly accurate results by co-precipitation of arsenic and antimony with iron. The author prefers to remove copper from the solution by electrolysis, and to titrate the arsenic in the exhausted electrolyte after suitable treatment. Two 10 grm. portions of the metal are dissolved in a slight excess of nitric acid, 17 c.c. of sulphuric acid are added to the contents of each beaker, and the whole evaporated to dryness. If selenium or tellurium is present, the copper sulphate is dissolved in water, the solution heated to 40° C., and a rapid current of sulphur dioxide passed through it. The solution is then filtered from any separated selenium or tellurium, acidified with 12 c.c. nitric acid, brought up or down to 120 c.c. by addition of water or concentration, and electrolysed, preferably by the slow method described by the author (see following abstract). In absence of selenium and tellurium the copper sulphate is dissolved in 12 c.c. nitric acid and about 110 c.c. of water, and electrolysed. In very accurate work the deposited copper is redissolved in 14 c.c. nitric acid (sp. gr. 1·42), 20 c.c. sulphuric acid (sp. gr. 1·84), and 50 c.c. water, and the solution made up to 120 c.c. and again electrolysed. The small portions withdrawn from the electrolyte, to determine by testing with hydrogen sulphide whether all the copper has been removed, are treated with a crystal of potassium bisulphite and a few drops of sulphuric acid, and evaporated to decompose the sulphides. The resulting solution is added to the exhausted electrolyte, which is next treated with 3 grms. of potassium bisulphite, and evaporated until the nitric acid is removed and the sulphuric acid has fumed for five minutes. In absence of more than traces of antimony, the liquid is next freed from the last traces of nitric acid by the method described later. If antimony must be separated, the solution is diluted with 1½ vols. of water, 2½ vols. of strong hydrochloric acid are added, and the arsenic separated in the cold by hydrogen sulphide. The arsenic sulphide is dissolved in fuming nitric acid, and when the sulphur has disappeared, 3 grms. of potassium bisulphite and 25 c.c. of strong sulphuric acid are added, and the solution evaporated until fumes of sulphuric acid have been in evidence for five minutes. To remove the last trace of nitric acid, the cooled solution is washed into a long-necked Kjeldahl flask and again evaporated, until the acid has fumed for five minutes. The neck of the flask is then heated, to drive out any condensate which may contain traces of nitric acid. The acid solution is made slightly alkaline by addition of ammonia, and then very faintly acid, after which 5 grms. or more of sodium bicarbonate and 3 drops of a 10 per cent. solution of potassium iodide are added, and the solution titrated with very dilute iodine solution (1 c.c. = 0·001 grm. As) and starch. With the precautions described for eliminating the last traces of nitric acid, the method is shown to be more accurate than any gravimetric method for the estimation of arsenic in copper. When the quantity of arsenic present is less than 0·05 per cent., two 10 grm. assays are combined, but for quantities less than 0·01 per cent. the error introduced by a trace of platinum, which always finds its way into the electrolyte, is appreciable, and tends to make the results a little high. G. C. J.

Exact Electrolytic Assay of Refined Copper. 1. Standard Method. 2. Rapid Assay by Rotation of the Electrolyte in a Solenoid. G. L. Heath. (*J. Ind. Eng. Chem.*, 1911, 3, 74-78.)—*Standard Slow Method.*—Five grms. of drillings are dissolved in 40 to 60 c.c. of the acid mixture, described later, contained in a beaker 12 cms. high and 6 cms. in diameter, the mixture being heated just short of the boiling-point. The solution is brought up to 120 c.c. by addition of water, and is electrolysed, using as cathode a plain foil cylinder 5 cms. high and with a total area (counting both sides) of 150 sq. cms. A split cylinder is preferred on account of ease of manipulation. The assays are started overnight with a current of 1½ ampères (1 ampère per 100 sq. cms.), and in the morning, as soon as the liquid is colourless, the split watch-glass covers are washed down and the current reduced to 0·5 ampère. Assays should finish in sixteen or eighteen hours without more than a trace of evolution of gas at the cathode. Otherwise they are best rejected, but it is possible to avoid oxidation or hydrogen absorption by reducing the current to 0·25 ampère, if, at the end of eighteen hours, deposition is not quite complete, and the cathode shows bubbles of gas. When deposition is complete, as shown by mixing 1 c.c. of the electrolyte with a few drops of hydrogen sulphide solution in a porcelain vessel, the stand supporting the beaker is withdrawn, the beaker replaced by one containing distilled water, and the electrode disconnected, washed with water and alcohol, and the last traces of the latter removed by burning. The acid mixture used for dissolving the copper is made up from 7 volumes nitric acid (sp. gr. 1·42), 10 volumes sulphuric acid (sp. gr. 1·84) and 25 volumes water. For the purest copper containing less than 0·03 per cent. arsenic, 40 c.c. of this solution is taken, with 0·03 to 0·1 per cent. arsenic, 50 c.c., and for copper containing 0·1 to 0·5 per cent. arsenic, 60 c.c. The results are accurate to within 0·01 per cent., providing the copper does not contain more than traces of antimony, selenium, or tellurium, in which case preliminary purification of the electrolyte is necessary. If there be doubt as to the freedom of the deposit from arsenic, it may be redissolved in 40 c.c. of the acid mixture, the solution diluted to 120 c.c. and the copper reprecipitated.

Rapid Assay by Rotation of the Electrolyte in a Solenoid.—The solenoid is constructed by winding 500 turns of No. 13 B. and S. gauge magnet wire upon a copper cylinder 7 cms. in diameter and 8 cms. high. The cylinder is brazed to two soft steel plates, 14 cms. in diameter and 2 mm. thick, the whole forming a spool, on which to wind the wire. The centre of the upper steel plate is cut out so that only a flange remains, and a 1-inch hole is also bored in the lower plate to provide for ventilation. The bottom plate carries three small knobs to support the beaker. The wire coil is conveniently put in series with the assay, and a current of 4·5 ampères is found most satisfactory. With this ampèrage there is no necessity to reduce the current towards the end of the assay, and it suffices for the deposition of 5 grms. of copper in two and a half hours. The cathode must be removed as soon as the last trace of copper is removed. It is necessary to complete success with the rapid method to use a gauze cathode of the general dimensions given for the foil cathode, but constructed of gauze with about 40 meshes per inch. Such an electrode, with edges suitably bound, weighs about half an ounce. Not more than 50 c.c. of the acid mixture must be used, or the heat and strong current will finally cause the metal to redissolve from the cathode. The results are as accurate as those given by the slow method. G. C. J.

Efficiency of Borax-Bead Tests for Nickel and Cobalt in Admixture.

L. J. Curtman and P. Rothberg. (*J. Amer. Chem. Soc.*, 1911, **33**, 188-189.)—The authors have determined the reliability of the borax-bead test in detecting the presence of nickel in admixture with cobalt, and *vice versa*. Solutions of the nitrates of these metals in varying proportions were mixed, and the sulphides completely precipitated from ammoniacal solutions. Portions of the freshly precipitated sulphides were fused in borax beads of approximately uniform size, and the beads viewed in daylight after treatment in both the oxidising and the reducing flames.

The results obtained were exactly similar when precipitated nickel and cobalt hydroxides were employed.

The results show that 1 part of cobalt can be detected in the presence of 30 of nickel, in which case a blue bead is obtained in the oxidising flame, and a bead of uncertain violet colour in the reducing flame. Proportions of Ni and Co from 20 : 1 to 1 : 1 gave in each case, both in oxidising and reducing flames, a blue bead. If the ratio of Ni to Co be increased, uncertain results are obtained till the proportion is 50 : 1, when a decidedly brown bead shows the presence of nickel, though, of course, 1 part of Co in 50 Ni is not shown by this reaction. Borax-bead tests can therefore only be relied on when the results are affirmative. A. R. T.

Bismuth Ochres from California. W. T. Schaller. (*J. Amer. Chem. Soc.*, 1911, **33**, 162-166.)—From analyses of the bismuth ochres found at several mines yielding lithium minerals in San Diego County, Colorado, the author is of opinion that the existence of natural Bi_2O_3 is not established, and that natural "bismite" or bismuth ochre, when pure, is more probably a hydroxide of bismuth. The bismuth ochres examined in this instance are either bismuth hydroxide or bismuth vanadate (pucherite), or mixtures of these two substances.

The analyses are as follow:

		I. Yellow Ochre (Stewart Mine).	II. Yellow Ochre (Pala Chief Mine).	III. Grey Ochre (Stewart Mine).
	Bi_2O_3	64·43	66·14	64·90
	V_2O_5	12·11	25·80	0·80
Gangue	Soluble in HNO_3	2·27	—	9·50
	Insoluble in HNO_3	17·63	7·37	13·50
H_2O	at 107° C.	0·32	0·21	0·40
	at 240° C.	0·24	0·32	0·30
	on ignition	3·43	0·84	11·40
		100·43	100·68	100·80

Remarks.—I. Mixture of vanadate and hydroxide of Bi. II. $Bi_2O_3.V_2O_5$ (amorphous "pucherite"). III. $Bi_2O_3.3Aq$. Vanadium an impurity from adhering yellow ochre.

The method of analysis was as follows: After ignition, the residue was dissolved in nitric acid, and the solution precipitated by hydrogen sulphide, the bismuth being converted into carbonate, and weighed as Bi_2O_3. The filtrates were evaporated to dryness, and the vanadium separated from the residue by a current of dry hydrochloric acid gas, the oxychloride of vanadium formed being collected in water, reduced and titrated with permanganate, after evaporating the chloride with sulphuric acid. The vanadium-free residue consisted chiefly of silica, alumina, and lime.

A. R. T.

Estimation of Very Small Amounts of Calcium by Means of Permanganate. L. T. Bowser. (*J. Ind. Eng. Chem.*, 1911, 3, 82-84.)—To the solution to be tested, about 5 or 10 c.c. in volume, a few drops of ammonia and 0·4 grm.

ammonium chloride are added. The mixture is heated to boiling, and 0·2 grm. ammonium oxalate is added. Boiling is continued for a moment, and the solution is then diluted with its own volume of 3 per cent. ammonia and allowed to stand two or three hours. The precipitate is collected on asbestos (previously treated with permanganate and washed), and washed with 3 per cent. ammonia, preferably hot. The asbestos pad is then transferred to a beaker, mixed with a little water, and, after addition of 1 c.c. of sulphuric acid (1 : 1), the mixture is heated nearly to boiling, and a slight excess of permanganate is added, the excess being determined by titration with oxalic acid. The solutions of permanganate and oxalic acid used are only $\frac{N}{200}$, and must be standardised against a calcium solution of known strength, for such highly dilute solutions are said to correspond to much less calcium than is indicated by theory. Narrow burettes are used, and it is shown that quantities in excess of 0·3 mgrm. of calcium can be estimated with an error not exceeding 0·03 mgrm. Quantities less than 0·3 mgrm. tend to be overestimated, and, accordingly, very weak solutions should be concentrated before applying the method. G. C. J.

Application of "Cupferron" in Quantitative Analysis. R. Fresenius. (*Zeitsch. anal. Chem.*, 1911, **50**, 35-43.)—The list of metals which can be separated from iron by precipitation of the latter by means of "cupferron" (the ammonium salt of nitrosophenyl-hydroxylamine, *cf.* ANALYST, 1910, **35**, 327) is now extended to include manganese, cobalt, zinc, and the alkaline earth metals. The separation of iron and alumina can be carried out more conveniently and with greater certainty of obtaining accurate results by this than by any other gravimetric method. The method serves well for the estimation of iron in ferromanganese or in manganese ores, but with ores the estimation of the manganese in the filtrate is more troublesome than is the similar estimation after an acetate separation, when this is conducted by an experienced operator.

The results obtained by Biltz and Hödtke with copper (ANALYST, *loc. cit.*) are confirmed, but bismuth is added to the list of metals which must not be present. Not only may iron and copper be separated from other metals by means of the reagent, but it may be used to separate iron from copper. To the acetic acid solution of the metals ammonium acetate is added and then excess of cupferron. The precipitate is washed with ammonia until the filtrate is no longer blue, and finally with water. In the filtrate the copper is thrown down by addition of acetic acid and a little more cupferron to render the precipitation complete. The copper precipitate is then treated as directed by Biltz and Hödtke. Small quantities of iron may be overestimated by as much as 5 per cent., but, in the absence of metals which interfere, copper can be estimated with an error not exceeding +0·2 per cent. G. C. J.

Quantitative Estimation of Lithium. E. Murmann. (*Zeitsch. anal. Chem.*, 1911, **50**, 171-174.)—The separation of lithium from sodium and potassium by precipitation as phosphate is apt to give high results, owing to the inclusion of impurities in the precipitate. The best procedure for carrying out this method consists in neutralising the solution of mixed chlorides and evaporating to dryness in a quartz dish with a few crystals of sodium phosphate. After cooling, the residue is extracted with dilute ammonia solution (1 : 1) and the insoluble phosphate is collected, dried and incinerated. The evaporation and filtration should be performed at least twice, if a substantial loss is to be avoided. The method yields fairly good results under favourable conditions, but there is a tendency on the one hand to incomplete precipitation of the lithium phosphate, and on the other hand to an accumulation of impurities if the precipitation be repeated frequently. The application of a correction for insoluble impurities is unsatisfactory.

The author has investigated an alternative method based on the extraction of lithium chloride by organic solvents. With anhydrous pyridine, lithium chloride may be separated with satisfactory results from sodium and potassium chlorides. The separation is very sharp; but, since the crystals of the other chlorides may retain traces of lithium chloride in their interior, it is perhaps advisable to redissolve the residue from the first extraction with pyridine, evaporate and extract a second time. The pyridine extract is evaporated to dryness in a platinum dish, the lithium chloride is treated with a slight excess of $\frac{N}{1}$ sulphuric acid, dried and heated to dull redness, and weighed as sulphate. If the latter be slightly coloured, the sulphate may be dissolved in water and a correction applied for the insoluble matter. In evaporating the solution of mixed chlorides for weighing, it must be borne in mind that lithium chloride is volatile at a low red heat. J. F. B.

Criticism of Magnesia Method for Estimation of Phosphoric Acid. W. C. Dumas. (*Chem. Engineer*, 1910, **12**, 185-190.)—The criticism is confined to methods in which the phosphoric acid is first separated by means of molybdate. It is shown that the composition of the precipitate most closely corresponds to the formula $MgNH_4PO_4.6H_2O$, when the precipitation is made slowly in solutions which are nearly neutral. After fifteen minutes a considerable quantity of ammonia is added and the process continued as usual, but the presence of even 1 c.c. of ammonia (sp. gr. 0·96) in each 50 c.c. of liquid at the moment of addition of magnesia mixture considerably reduces the percentage of magnesia in the precipitate. Rapid addition of magnesia mixture (6 or 7 drops per second) gives rise to a precipitate containing more than the theoretical amount of magnesia. Precipitates of abnormal composition behave abnormally on heating in a blowpipe flame, but even those of normal composition after the first five minutes' heating may continue to lose weight at a nearly uniform rate of 0·1 mgrm. per two minutes for half an hour or more. A protest is raised against the use of "analysed" samples as controls. The author recommends the use of pure silver phosphate for purposes of control. The silver phosphate is dissolved in nitric acid, the silver precipitated as chloride, and the phosphoric acid in the filtered solution precipitated by means of magnesia mixture after addition of as much molybdic acid as would be present in an actual determination, followed by addition of ammonia to very faint alkaline reaction. The use of molybdic acid in the control experiment is advised because, in presence of molybdic acid, precipitates of abnormal composition are less likely to be obtained.

G. C. J.

Electrolytic Estimation of Manganese. J. Köster. (*Zeitsch. Electrochem.*, 1911, **17**, 57-58.)—Otin's criticism (ANALYST, 1909, **34**, 415) of Engels' method for the estimation of manganese is based on a misreading of the directions of Engels, who specified the addition to the electrolyte of 10 grms. of ammonium acetate—not sodium acetate—together with 2 grms. of chrome alum. With the use of this electrolyte current densities as high as 2 to 2·6 ampères per 100 sq. cm. of anode surface do not lead to the deposition of even 0·1 mgrm. of manganese on the cathode. With such high-current densities a deposit does appear on the cathode, but this is not manganese, but chromium. Repetition of Otin's experiments, using sodium in place of ammonium acetate, showed that the cathode deposits observed by him were mainly chromium, with, however, about 1 mgrm. of manganese peroxide.

The one objection to Engels' method is the risk of chromic acid adhering to the deposit on the anode, even when this is washed with hot water after ignition, and the author therefore prefers to avoid the use of chrome alum, and to conduct the electrolysis in presence of alcohol, with high-current densities and mechanical agitation (*cf. Zeitsch. Electrochem.*, 1904, **10**, 553). G. C. J.

Separation of Platinum and Tin. L. Wöhler and A. Spengel. (*Zeitsch. anal. Chem.*, 1911, **50**, 165-171.)—Owing to the close similarity of their chemical functions and the formation of complex ions, the separation of platinum and tin presents considerable difficulties. Tin cannot be extracted from platinum-tin alloys by means of acids, reducing agents afford no separation from solutions, and the sulphides cannot be separated either by concentrated hydrochloric acid or by sodium hydroxide. A practicable but tedious method of separation may be based on the volatilisation of stannic sulphide in an atmosphere of sulphuretted hydrogen, or on the volatilisation of stannic bromide in a current of bromine vapour, which is preferable to chlorine for this purpose. The hydrolytic decomposition of the platinum stannic compounds by sulphuric acid only affords a complete separation if conducted at a temperature of 150° to 200° C. in a closed tube; at temperatures below 100° C. a certain amount of platinum is generally absorbed by the colloidal stannic oxide. The simplest and most accurate separation is obtained by precipitation of the stannic acid by heating with dilute sulphuric acid, and subsequently depositing the platinum by electrolysis from the clear liquid over the precipitate of stannic acid. For instance, a solution was prepared, containing 1 grm. of platinum and 3 grms. of tin, in 25 c.c. each of nitric and hydrochloric acids, subsequently diluted to 500 c.c. Fifty c.c. of this solution were treated with 25 c.c. of $\frac{N}{2}$ sulphuric acid, and the stannic acid was precipitated by boiling. After standing for twenty-four hours the liquid was electrolysed, with a tension of 1·1 to 1·7 volts, $N_{D100} = 0·2$ ampère, either at the ordinary temperature or at 50° to 60° C. The completion of the reaction was indicated by the decreased yellow coloration of the liquid when tested with stannous chloride. During the electrolysis the precipitate should not be disturbed, but at the end occasional stirring is necessary to complete the separation. Subsequently the stannic acid is converted into a flocculent condition by the addition of ammonium nitrate, and filtered off. J. F. B.

Estimation of Potassium as Potassium Platini-Chloride. H. Fresenius and P. H. M. P. Brinton. (*Zeitsch. anal. Chem.*, 1911, **50**, 21-35.)—A study of the influence exercised on the results of this determination by notable amounts of calcium or magnesium chloride when these are not first separated. The experiments were all made with approximately equal amounts (0·5 grm.) of potassium chloride and the other chloride. In presence of its own weight of calcium chloride, 0·5 grm. of potassium chloride is overestimated by no more than 0·5 per cent., and this error may be reduced to 0·3 per cent. by re-treatment of the weighed precipitate with alcohol, filtering, etc. The disturbing effect of magnesium is less—not exceeding 0·3 per cent. The influence of varying strengths of alcohol was also tried, and the use of the customary 80 per cent. spirit is confirmed as most satisfactory. Weaker alcohol leads to low results, even 70 per cent. spirit reducing the results nearly 1 per cent. The use of very strong alcohol may lead to high results in presence of sodium platini-chloride, which may be partially reduced, the resulting platinum getting weighed as potassium platini-chloride, but the error due to the use of alcohol as strong as 95 per cent. is negligible. The authors separate their precipitate from the filter, wash the latter with hot water, evaporate the filtrate in a tared dish, add the precipitate, dissolve this in hot water, re-evaporate and dry at 130° C. Constant weight is attained in two hours. If bulky precipitates are not dissolved in the dish, to ensure their distribution in a thin layer, water will be retained even after sixteen hours' drying, and may give rise to results 0·2 to 0·3 per cent. too high. G. C. J.

Dissolving Silicon Alloys. G. C. Davis. (*J. Ind. Eng. Chem.*, 1911, **3**, 42-43.) —It is found that 50 per cent. ferrosilicon can be readily decomposed by a mixture of strong hydrofluoric acid with hydrochloric or sulphuric acid. Solution is complete in a few minutes at a low temperature, and it is not necessary to reduce the sample beyond 10 mesh. Silico-spiegels with 20 per cent. silicon, 40 per cent. manganese, and 40 per cent. chromium may be similarly decomposed. G. C. J.

New Method of Estimating Sulphuric Acid and Sulphates. V. Auger and M. Gabillon (*Comptes rend.*, 1911, **152**, 441-443.)—The method is based upon the reduction of the sulphuric acid or sulphate by means of hydriodic acid, and the volumetric estimation of the liberated hydrogen sulphide by means of a standard solution of iodine. The hydriodic acid is prepared in the flask in which the estimation is made by the interaction of a mixture of phosphoric and pyrophosphoric acids with potassium iodide. The iodine liberated in the reaction of the sulphuric acid with the hydriodic acid is reconverted into hydriodic acid by the addition of phosphorous acid. The hydrogen sulphide produced in the reaction

$$H_2SO_4 + 8HI = H_2S + 4H_2O + 8I$$

carries forward some iodine and hydriodic acid, the first of which is eliminated by passing the gas through a column of calcium iodide, and the latter by means of an aqueous solution of the same acid decolorised by the addition of phosphorous acid. Finally, the hydrogen sulphide is absorbed in a solution of zinc acetate, and the sulphide estimated by means of iodine in the usual way. The reaction begins at about 80° C., and finishes at 110° C., and is complete in twenty to twenty-five minutes. To obtain good results, it is essential that the mixture of phosphoric acid used shall be free from metaphosphoric acid (which would cause too energetic reduction of the phosphorous acid, with the liberation of red phosphorus, etc.), and that the reduction flask shall be free from moisture so as to prevent the hydrogen sulphide being oxidised by the iodine into sulphur and hydrogen iodide. The apparatus required for an estimation consists of a 50 c.c. flask, provided with a long neck, with a lateral tube, through which the gas escapes, and with means for the introduction of a tube for the admission of dry carbon dioxide throughout the operation. The lateral tube is connected with a glass tube, about 25 cm. long and 1 cm. in diameter, charged with iodine for a length of 3 cm., and then filled up with calcium iodide. Attached to the other end of this tube is a small washing flask, containing about 5 c.c. of hydriodic acid solution, whilst the apparatus is completed by two absorption vessels, together containing about 100 c.c. of zinc acetate solution (25 grms. per litre). The reduction flask is charged with about 20 grms. of phosphoric acid (obtained by diluting the acid of 60° Bé. to five-sixths of its volume), 2 grms. of phosphorous acid, 3 grms. of powdered potassium iodide, and about 0·2 grm. of the finely-powdered sulphate under examination. As soon as all air has been expelled from the apparatus by the current of carbon dioxide, the flask is heated in an oil or sulphuric acid bath. The method gives results closely agreeing with theory, and is applicable to all sulphates except barium sulphate, which is attacked too slowly to be analysed in this way. C. A. M.

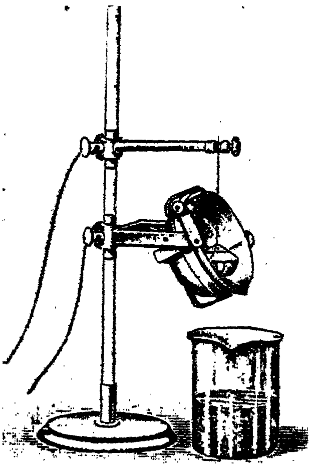
Estimation of Tellurium. A. Rosenheim and M. Weinheber. (*Zeitsch. anorg. Chem.*, 1911, **69**, 266-269.)—Gutbier's method for the estimation of tellurium (*Ber.*, 1901, **34**, 2724), by reduction with hydrazine in weakly alkaline solution, fails to throw all the tellurium out of solution, and yet gives results above the truth because the separated tellurium suffers oxidation during the prolonged boiling which is necessary to secure even approximately complete precipitation. By conducting the reduction in an autoclave, the method may be made more exact. To the solution contained in a Jena beaker, a sufficient quantity of a slightly ammoniacal 10 per cent. solution of hydrazine sulphate is added, and the beaker covered with a clock-glass, and placed in an autoclave, which is then heated up to about 130° C., and kept at this temperature for thirty minutes. The separated tellurium is collected in a Gooch crucible, washed with a very dilute solution of sulphur dioxide, and dried at 105° to 115° C. in a current of carbon dioxide. No trace of tellurium can be found in the filtrates. Oxidation of the precipitate is not wholly avoided, but the error on a weighing of 0·1 grm. never exceeds +0·7 mgrm., and is usually +0·5 mgrm.

Telluric acid can be titrated with fair accuracy like boric acid in presence of a sufficient quantity of some polyhydroxy compound such as glycerol. The aqueous solution of telluric acid is mixed with an equal bulk of glycerol (sp. gr. 1·26) and titrated cold with $\frac{N}{10}$ caustic alkali, using phenolphthaleïn as indicator. Each c.c. $\frac{N}{10}$ alkali = 0·0225 grm. H_6TeO_6, the glycerol-telluric acid reacting as a monobasic acid. The error of the method may be as great as ±3 per cent. on small readings of less than 15 c.c.

More accurate results can be obtained by addition of an excess of $\frac{N}{10}$ caustic soda containing some barium chloride, to a solution of telluric acid, and titration of the excess of caustic alkali by means of oxalic acid with phenolphthaleïn as indicator. Direct titration with barium hydroxide is not successful, as the end-point of the titration is indistinct, and barium tellurate is much more soluble in water than in solutions of barium hydroxide. On addition of excess of caustic soda containing barium chloride, barium tellurate separates almost completely, and the excess of alkali must be determined at once without filtration by titration in the cold with oxalic acid. Each c.c. $\frac{N}{10}$ alkali $= 0{\cdot}01125$ grm. H_6TeO_6, the telluric acid here behaving as a dibasic acid. The extreme error of the method is ±1 per cent. G. C. J.

Reaction of Uranium Salts. J. A. Slemssen. (*Chem. Zeit.*, 1911, **35**, 139.)—A solution of commercial ethylenediamine affords a sensitive reagent for uranium salts, which, when present even in traces, react with it immediately to form a bright yellow crystalline precipitate, which dissolves on adding an excess of the reagent. The test is quite as sensitive as the potassium ferrocyanide reaction and hydrogen peroxide test of Aloy. C. A. M.

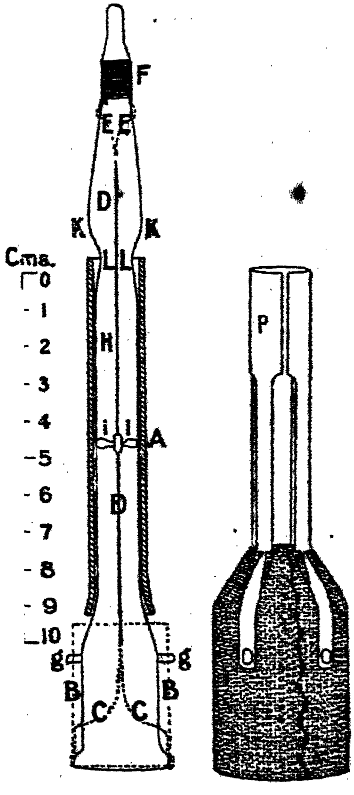
APPARATUS, ETC.



Holder for Classen Dish. F. Blume. (*Chem. Zeit.*, 1911, **35**, 12.)—The dish rests in a basket constructed of strips of nickel sheet instead of in the usual ring. This basket can be turned on a horizontal axis, and means are provided for holding it rigidly during the electrolysis proper. When this is at an end, the basket is tipped through 90° and the contents caught in a beaker. The arrangement permits of the use of very small quantities of wash-water—40 c.c. in place of 400 c.c.—a considerable advantage when the electrolyte and washings have to be used for the determination of other constituents. The apparatus is made by R. Hase, Hanover. G. C. J.

Gooch Crucible for Micro-Analysis. J. Donau. (*Monatsh.*, 1911, **32**, 31-40.)—The author and Emich (Analyst, 1910, **35**, 137) have described apparatus for filtering minute quantities of material. The author now describes a miniature Gooch crucible for use in the place of the paper discs previously described. From platinum foil of 0·0035 mm. thickness, a disc 15 mm. in diameter is punched. To the edge of this disc a platinum wire, 20 mm. long and 0·1 mm. thick, is welded. The disc is then laid on a piece of paper on a smooth glass plate, and 80 to 100 small holes are pricked in it by means of a fine needle, no holes being pricked within 3 or 4 mm. of the edge, which may be covered with an annular piece of paper. The disc is then reversed and laid on a soft rubber stopper, so that the burred side is uppermost, and a cylindrical piece of glass, brass, etc., with perfectly smooth table end, about 8 mm. in diameter, is pressed firmly against the pricked centre, and the disc given the form of a little dish 8 mm. in diameter and 3 to 4 mm. high. This dish is used as a Gooch crucible with the apparatus before described, its under surface just fitting the capillary funnel of Emich and Donau. In the ordinary way it is packed with about 0·5 mm. of asbestos, free from dust or long fibre, but it may be converted into a Munroe crucible by heating in it ammonium platinichloride, pressing, repeating the charge of platinichloride, and repressing. The attached platinum wire is for convenience in manipulation, ignition, weighing, etc., and is usually bent into a hook. G. C. J.

Apparatus for Rapid Electro-Analytical Determination of Metals. H. J. S. Sand. (*Chem. News*, 1911, **103**, 14-15.)—The apparatus has been designed to retain as far as possible the features of the apparatus previously described by the author (*J. Chem. Soc.*, 1907, **91**, 373; 1908, **93**, 1572) whilst reducing the use of platinum to a minimum. The anode is made largely of glass, the cathode of silver or nickel. The wide glass stem of the inner electrode (left in cut) rotates inside the glass tube A, which latter is gripped by the collar, P, of the outer electrode. In the inner electrode, platinum is only used in the gauze cylinder B, in the strips of gauze, C, which lead into the interior of the electrode to the copper wire, D, and in the strips, E, leading out at the top, which are wound over by the platinum wire F. All the connections through the glass are made by strips of platinum gauze of threefold thickness at the top, and with a width not less than 1 cm. below, so that it is possible to pass currents up to 10 ampères without endangering the electrode. The gauze cylinder, B, is fitted to the bottom of the electrode by blowing the glass against it. At the top, the cylinder stands off from the glass by 2 to 3 mm. in order to allow the gas bubbles to escape readily. It is held securely by four small protuberances, *g*, on the glass. The copper wire is held in the interior of the tube H by the enamel support *i*. The stem is widened at K just enough to prevent the tube A from being pulled off, but not enough to hinder the collar, P, from being slipped over it. The contraction of the stem at L allows the space between the stem and A to be washed down readily with a few drops of water before disconnecting.



For copper determinations a cathode of silver is employed, and the deposits are removed at the end of an experiment by means of hot dilute sulphuric acid. In constructing the cathode, a frame is cut from a single piece of metal sheet about 0·5 mm. thick and bent into the shape shown in the unshaded portion of the right-hand sketch. A cylinder of Kahlbaum's coarse gauze is then constructed, and the final form is obtained by introducing eight small pleats at the top. Gauze and frame are held together by four stout rivets, of which two are shown. Silver being brittle, loose ends are best avoided by removing a few horizontal wires at the bottom and melting the end of the projecting vertical wires in the oxygen blowpipe. The vertical joint is made by removing a few vertical wires, twisting together the opposite horizontal threads and fusing. Nickel cathodes for zinc determinations are made in a similar way from gauze of 11 meshes per cm., but of course no attempt is made to fuse the ends of the wires, nor is this necessary with nickel on account of its superior mechanical properties. G. C. J.

Electrolytic Estimation of Copper and Zinc using Sand's Apparatus.

W. M. Smalley. (*Chem. News*, 1911, **103**, 15-16.)—The apparatus used was that described by Sand (see preceding abstract). Copper was deposited from warm solutions containing only very small amounts of nitric acid (0·1 to 0·5 c.c.) to minimise the difficulty of washing the deposits without loss. About 0·7 c.c. of sulphuric acid was contained in the 25 to 75 c.c. of solution operated on. A current of 10 ampères was employed so long as the solution remained visibly blue, it was then reduced to 5 ampères until the ferrocyanide test showed the liquid to be free from copper, and finally increased to 10 ampères during the process of disconnecting. Providing the amount of nitric acid did not greatly exceed 0·1 c.c., quantities of copper up to 1 grm. could be deposited in 10 minutes with an error in no case exceeding −0·4 mgrm. With as much as 0·5 c.c. nitric acid present, the error may reach −0·6 mgrm.

Zinc was determined with the apparatus, following the usual procedure, the results being equal to those yielded by platinum apparatus. It was found that the use of citric acid in place of acetic acid reduced the tendency to occasional high results, but the improvement was small and the time required for deposition was nearly doubled.

A black deposit which forms on the silver electrodes may be removed as far as possible with a silk handkerchief, but it is not advisable to remove it entirely by ignition, as this leaves the surface in a porous state, in which it is more readily attacked by ozone in subsequent determinations. G. C. J.

Errors in Determining Sizes of Mineral Grains and the Use of Surface Factors.

H. E. Ashley and W. E. Emley. (*J. Ind. Eng. Chem.*, 1911, **3**, 87-91.) —It is inaccurate to express the size of microscopic mineral grains in "diameters," unless the term is carefully defined. The volumes calculated from microscopic measurement require multiplication by correction factors. For example, about 100 particles of sand were weighed, and their volume calculated by dividing this weight by the sp. gr. of quartz. The particles were then measured in two directions by means of an eye-piece micrometer, and, as they appeared on plan to approximate a rectangular rather than a circular shape, their volume was calculated from the formula $n(l \times b)^{\frac{3}{2}}$, where l represented the average length, b the average breadth, and n the number of the particles. The use of this formula overestimated the volume of the particles more than twofold in one case, and more than threefold in another. Calculation of the volume from the apparent mean diameter, on the assumption that the particles approximated a spherical shape, would have given a nearly correct value in the first case, but still one much in excess of the truth in the second, and the particles bore no resemblance to spheres. Obviously their depth was much smaller than their horizontal dimensions, and with kaolin or a micaceous sand the error would be even greater.

The distribution of sizes in any fraction of clay obtained by mechanical analysis may follow several laws; hence the various formulas proposed for calculating the mean size are all liable to error. A simple empirical correction factor to the arithmetical mean is, apparently, as accurate as any more elaborate calculation. Mellor's (*Trans. Eng. Cer. Soc.*, 1910, **9**, 94) surface factor, $\frac{6}{s}\left(\frac{W_m}{d_m}+\frac{W_n}{d_n}+\frac{W_o}{d_o}+\ldots\right)$, or Purdy's simpler form, $\frac{W_m}{d_m}+\frac{W_n}{d_n}+\frac{W_o}{d_o}+\ldots$, best represent the results of a mechanical analysis.

The surface factors become incomparable if different size limits are used in calculations for the smallest sizes. It is advocated that Seger's limits—0·04, 0·025, and 0·010 mm.—give results of most value to clayworkers. Other workers (*e.g.*, Purdy, *Illinois Geol. Survey Bull.*, **9**, 152) have subdivided the finest group into particles above and particles below 0·001 mm. mean diameter; but this gives predominance to the finest group, which may only amount to 10 per cent. of the whole, and yet accounts for 80 per cent. of the calculated surface, which then comes out very high. There is a doubt whether these very fine particles retain their crystal condition or whether they do not pass over to the colloid state, and there is no doubt that for some ceramic purposes clays with only slight residues coarser than 0·01 mm. possess grave disadvantages. Hence the preference expressed for Seger's simpler grading.

G. C. J.

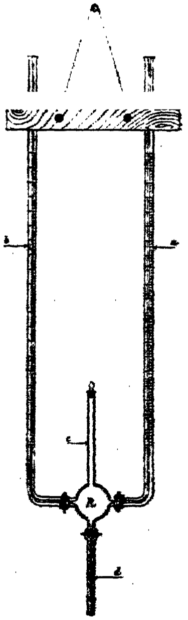
Determination of the Electrical Conductivity of Soils. J. König, J. Hasenbäumer, and H. Meyering. (*Zeitsch. angew. Chem.*, 1911, **24**, 103-106.)—For the determination of the electrical conductivity of soils, the authors make use of the telephone method, placing the soil between platinum electrodes in an ebonite box of special construction. The vertical electrodes are 10 cm. long by 5 cm. high, and are fixed 1 cm. apart in the box, which is just long enough and high enough to accommodate them, and 2·5 mm. broad. The bottom of the box is provided with numerous perforations and is covered with a sheet of paper. The space between the electrodes is filled with air-dried, finely divided soil, the box stood in a glass vessel containing conductivity water, and the whole placed in a large desiccator containing dilute sulphuric acid and caustic potash. When moisture is seen to have risen to the surface of the soil, the box is left one hour longer in the desiccator, dried and weighed. It is then returned to the pan of water in the desiccator for a few hours or overnight and reweighed. When the weight is constant, the box is transferred to a rectangular glass vessel just large enough to accommodate it, and this is placed in a water-bath at 18° C. for at least an hour. Both the rectangular glass vessel and the water-bath have covers provided with holes through which the terminals of the electrodes project. The conductivity is then determined in the usual manner, and compared with that of a $\frac{N}{100}$ solution of potassium chloride contained in the same cell. Before use the electrodes are coated with platinum black by immersion in a solution containing 3 grms. platinum chloride and 0·02 to 0·03 grms. lead acetate in each 100 c.c.

The specific conductivity of soils is of the order $10^{-4} - 10^{-3}$, and experiments are described which show that it is reduced by cropping, in some cases by as much as 50 per cent., confirming the view that the conductivity depends in a great measure on the presence of those substances which serve as food for vegetation. The soil of several experimental plots was examined as to its conductivity, and the results compared with the weights of dry produce per acre. The soil of highest conductivity (97×10^{-5}) gave the largest yield, that of lowest conductivity (14×10^{-5}) the lowest yield. The intermediate members of the series did not grade well, but it is held that the conductivity of a soil is a fair index to its fertility, and a more certain index than the "osmotic water absorption" (*cf.* König and Hasenbäumer, *Zeitsch. angew. Chem.*, 1909, **22**, 1009), though in most cases the conductivity, osmotic water absorption and fertility (judged by weight of produce) rise and fall together. G. C. J.

Application of the Osmotic Pressure and Electrical Conductivity in the Examination of Soils. J. König, J. Hasenbäumer, and J. Meyering.

(*Landw. Vers. Stat.*, 1910, **74**, 1-56; through *Chem. Zentralbl.*, 1911, **1**, 259.)—In the estimation of the osmotic water-absorption capacity of a soil, the discrepancies formerly encountered may be almost entirely avoided by carrying out the determinations at a constant temperature. A temperature of 18° C. was found to be most favourable. Instead of estimating the osmotic absorption capacity, the soluble constituents of the soil may be measured by determinations of the electrical conductivity of the soil. This method is simpler and more rapid. But, on the other hand, the determination of the osmotic water-absorption capacity, given an efficient osmometer, appears to have the advantage of greater certainty and accuracy. The values found by both methods are satisfactorily concordant. The conductivity was determined by Kohlrausch's method. Since the electrical conductivity increases with the degree of fineness of the soil, it is advisable in comparative tests to use soils of the same degree of fineness. By these methods the authors have investigated the adsorption of nutrient salts by soils. The salts were employed at $\frac{N}{50}$ concentration for di-ionic salts and $\frac{N}{75}$ for tri-ionic compounds and potassium and acid calcium phosphates. The soils moistened with these solutions showed increases in the above characters, except in the case of dipotassium hydrogen phosphate, a circumstance which shows that with this salt both the potassium and the phosphoric acid are completely adsorbed by the soil. Other problems in the chemistry of soils have also been studied by means of the methods described above. J. F. B.

Volumetric Analysis with Small Volumes of Liquid. F. Pilch. (*Monatsh.*, 1911, **32**, 21-29.)—By means of the apparatus figured, very small quantities of the order of 1 mgrm. may be estimated with an error not exceeding 0·5 per cent. The reactions take place in the bulb *R*, which has a capacity of about 20 c.c. The burettes *a* and *b*, which are used to deliver any $\frac{N}{100}$ solutions appropriate to the purpose, are about 40 cm. long and 3·5 mm. in internal diameter. Iodeosin as recommended by Mylius and Förster (*Ber.*, 1891, **24**, 1482) is preferred as an indicator. Experiments are described showing that the accuracy obtainable is as stated, whether the apparatus be applied to alkalimetry, iodometry, or to estimation of chloride by Volhard's method. The apparatus has also been applied to nitrogen estimations by Kjeldahl's method. In this case, the ammonia is distilled from a small (100 c.c.) flask through a wide (1 cm.) glass tube bent obtusely, so that any products of the action of steam on the glass may flow back with condensed water to the flask. The vertical portion of this wide tube is packed with broken porcelain, and is drawn out to smaller bore at its lower end, which pierces the cork of the distillation-flask. From the upper end of the obtusely-bent tube, which carries a rubber cork, the vapours of ammonia, together with steam, are led through an acutely bent platinum tube of 5 mm. bore into the bulb, *R*, of the apparatus. The upper part of the vertical limb of the platinum bend carries a small (5″) condenser. By the use of a Nernst micro-balance 1 mgrm. or so of a substance of which very little is available may be weighed out accurately, and a sufficiently accurate nitrogen determination made in the manner described. G. C. J.



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REPORTS.

Vitality of Typhoid Bacilli in River Water, with Special Reference to the Question of Storage. A. C. Houston. (Metropolitan Water Board. Sixth Research Report, 1910.)—Previous experiments conducted by the author (ANALYST, 1908, **33**, 403) had shown that typhoid bacilli perished fairly rapidly in stored river water. The water was infected with the bacilli and allowed to stand in the laboratory, when 99·9 per cent. of the bacilli perished in one week, and all of them in nine weeks. The bacilli used were strains cultivated in the laboratory in the ordinary way, and it did not follow necessarily that uncultivated bacilli—that is, those obtained directly from the human subject—would perish in the same manner. An opportunity recently arose of performing the experiment with the uncultivated bacilli. The urine from a carrier case of typhoid fever, which was swarming with typical living bacilli, was centrifuged, and the deposit added to 5 litres of raw Thames water in a partially filled bottle which was stored in the dark at 64-67° F. At the commencement of the experiment the infected water contained 770,000 typhoid bacilli per c.c. At the end of one week these had been reduced to 4 per c.c. in the shaken sample, at the end of two weeks none could be detected in 100 c.c., and the same result was obtained on subsequent occasions for thirty-four days. On the 24th and four following days a half-pint of the shaken sample was drunk by the author without any ill effects. A second experiment with a fresh sample from the same case was made. The water after infection contained 1,650 typhoid bacilli per c.c., and at the end of one week there were none present in 100 c.c. In this experiment, also, a half-pint of the water was drunk on the twenty-third day from the start, and the four following days, without ill-effect.

A comparable experiment was made with bacilli from the same source, but cultivated in the laboratory for nine days on three changes of media before being added to the water. After one week's storage the number of typhoid bacilli in the infected water had been reduced from 15,800 to 200 per c.c., but it was not until after six weeks that they completely disappeared from the water.

It is thus seen that the uncultivated bacilli perish more quickly than the cultivated bacilli in the stored water, so that the conclusions previously expressed as to the efficacy of storage are confirmed, and their value enhanced by these experiments. As it is the uncultivated bacilli which naturally gain access to water, it is clear that even one week's storage of the raw river water is an enormous protection against the conveyance of typhoid fever, and that less than a month's storage would be apparently an absolute protection.

The methods employed were the same as in the previous report, but full details of the composition of the culture media used are given. J. H. J.

Report on the Solubility, Extraction, and Reactions of Certain Colouring Matters used in Foods. H. M. Loomis. (*U.S. Department of Agriculture, Bureau of Chemistry, Circular No.* 63, January 17, 1911, pp. 1-69.)—Although no report can nclude all food colouring matters, yet the greater number of the natural and coal-tar colours usually employed are here mentioned, and their reactions described. The report consists in the main of a number of comprehensive tables, comprising (1) determinations of the solubility of the colouring matters, in various solvents, by weighing the residue obtained on evaporation of the saturated solutions of the colours; (2) estimations of the amounts of colouring matter extracted by immiscible solvents in neutral, alkaline and acid solutions; (3) and (4) observations of the colour reactions of dyed fibres obtained from 0·5 per cent. solutions (of coal-tar colours), and 5 per cent. solutions (of natural colours), and of the colouring matters in aqueous solution, and with the dry colour and sulphuric acid before and after dilution. A few details of the methods of manipulation with regard to the examination of oils and fats, the detection of mixed colours, and the methods of mordanting wool are also given. A. R. T.

Extracts from the Proceedings of the Association of Official Agricultural Chemists, 1910. (*U.S. Department of Agriculture, Bureau of Chemistry, Circular No.* 66, 1911, 1-27.)—In this circular are given the various methods of analysis and modifications of methods which have been investigated and adopted by the association. The methods relate to the analysis of soils, inorganic plant constituents, insecticides, waters, dairy products, foods and feeding stuffs, sugar, tannin, medicinal plants and drugs, etc. W. P. S.

REVIEWS.

ALLEN'S COMMERCIAL ORGANIC ANALYSIS. Fourth Edition. Vol. III. "Hydrocarbons," by F. C. Garrett, D.Sc.; "Bitumens," by S. S. Sadtler, S.B.; "Naphthalene and its Derivatives," by W. A. Davis, B.Sc.; "Anthracene and its Associates," by S. S. Sadtler; "Phenols," by S. S. Sadtler; "Aromatic Acids," by E. Horton, B.Sc.; "Gallic Acid and its Allies," by W. P. Dreaper; "Phthalic Acid and the Phthaleins," by W. A. Davis; "Modern Explosives," by A. Marshall. 1910. London: J. and A. Churchill. Price 21s. net.

Since the issue of the last edition of this work, the lamented death of the author and the rapid growth of subject-matter has necessitated the work of revision being divided amongst a number of specialists, each responsible for a section. The volume under notice contains a mass of information indispensable to the analyst, and it is evident that a great amount of labour has been spent on the work of revision, though some parts have been better done and more thoroughly brought up to date than others.

The opening chapter on "Hydrocarbons" has been condensed by the elimination of textbook matter, without the addition of much new material and without a thorough revision of the text. Under bromine absorptions the latest reference is to McArthur's paper in 1888; no mention is made of McIlhiney's work, nor of the fact that carbon tetrachloride is now generally used as solvent in preference to carbon disulphide. Data concerning commercial acetylene are still given from Fowler's paper in 1898, without any reference to the more recent Cantor Lectures and treatise on the subject by Lewes, and the later work of Caro and others. Later and more complete analyses of the gas should have been given than those on p. 9. Instructions for the assay of crude shale oil have been reduced to very meagre dimensions; and although coke oven tar has been rewritten as a separate section, and some useful additions made to the instructions for the assay of coal-tar and coal-tar pitch, also some analyses of water-gas tar, the information on the tars generally seems capable of being made more complete and useful.

In contrast to the foregoing, the chapter on "Bitumens" has undergone a thorough revision, and much new matter has been added, chiefly, however, from American sources, the pages having increased in number from 141 in the third edition to 206. In connection with the determination of sulphur in petroleum, reference might have been made to the method of Garrett and Lomax (*J. Soc. Chem. Ind.*, 1905, **24**, 1212), which is similar to that of Sadtler, was published independently at about the same time, and does not need a crucible of special pattern. The analytical methods for asphalts have been rewritten and brought up to date, the official methods of the Society (American) for testing materials, and those of Clifford Richardson, being given in full, and new sections added on asphalt fluxes and bituminous road materials; the analyses of crude land pitch and Trinidad Lake pitch printed at the top of p. 62 are, however, not intelligible, owing to the omission of the headings to the columns. Other serious printers' errors are the omission from p. 122 of the illustration of the Abel flash-point apparatus, described as Fig. 2, and from p. 155 of the Doolittle Torsion Viscometer, said to be shown in Fig. 6, which represents the Redwood Viscometer. There is a paucity of illustrations; the Pensky-Martens Tester, or the modified form introduced by Gray, essential for oils of high flashing-point should, we think, have been illustrated, and figures might have been given of the Elliott and Foster Testers and the Tagliabue Cup, used in the United States.

In the new section on gas oil it is surprising to find no reference to the important papers on the composition and valuation of gas oils by Ross and Leather, published in the ANALYST in 1906-7. Fuel oil is not mentioned. Much useful information is given on lubricating oils, though it would have been better if the term "viscosity" had not been used where "efflux time" or "relative efflux time" is meant. Reference might have been made to the method worked out by Archbutt and Deeley ("Lubrication and Lubricants") of standardising commercial viscometers with mixtures of glycerol and water so that the results may be expressed in absolute measure. A formula by Ubbelohde for calculating "specific viscosity" from Engler "degrees" is given on p. 173, but no reference is made to the original paper, and the particulars given are too brief to be of use; the meaning of "Engler degrees" does not seem to be anywhere explained. The paragraphs on "Inner Friction" contain some inaccurate and curious statements. The definition of "absolute inner friction," by which the reviser means "viscosity," is not correct in all its terms, and is not expressed in intelligible language. It is curious to read that "capillary tubes give somewhat truer values than a viscometer, such as the Engler." The fact is that capillary tubes of the dimensions necessary to insure steady viscous flow are alone capable of giving true values, and commercial viscometers, such as the Engler, having very short and comparatively wide jets, merely measure rates of flow which happen to be nearly proportional to the viscosities of oils not lower in viscosity than sperm oil at 60° F., but which are far from proportional in the case of more fluid oils and water. The application of the formula given on p. 174 to commercial viscometers would therefore be quite useless. Loss by heating, a most valuable test for cylinder and superheater oils, and for comparing together mineral oils from different crudes, is much more accurately and satisfactorily determined by passing a known current of air over a weighed quantity of the oil in a tube heated in an air-bath than by the crude method described on p. 159. A useful section has been added on Lubricating Greases, and the American methods of assay have been added to the article on "Benzene." The reference in note 2 at the bottom of p. 176 should be 170, and not 162.

L. ARCHBUTT.

Naphthalene and its Derivatives—Anthracene and its Associates—Phenols—Aromatic Acids—Gallic Acid and its Allies—Phthalic Acid and the Phthaleins.—The section on naphthalene and its derivatives includes a synopsis of the physical and chemical properties of this hydrocarbon and those of its derivatives which are employed as antiseptics. The picric acid method of estimating naphthalene is fully described, and due attention is given to the analytical reactions of α- and β-naphthol.

Anthracene is similarly dealt with, and the chemistry of the constituents of crude anthracene is briefly outlined. Reference is made to the compounds obtained from picric acid and these associates of anthracene, as in some instances the products afford a valuable means of identifying these hydrocarbons. The important Luck test for anthracene is given with full details, including the improvement in the method introduced by Messrs. Meister, Lucius, and Brüning.

The next section is devoted to a consideration of phenol and its homologues of the benzene series. In the descriptive portion of this chapter are included the processes commonly employed in manufacturing these substances. The analytical treatment is very full, and deals both with the characteristic reactions of the purified compounds and with the analysis of such commercial articles as crude carbolic acid, carbolic disinfectants, sheep-dips, etc.

The important and intricate group of substances included in the category of creosote and creosote oils is adequately dealt with, and many references are given to original communications bearing on this very involved branch of organic analysis.

Unfortunately, this section contains many typographical errors (see pp. 289, 308, 314, 315, 351, 383), and the terminology is not always consistent. On the same page (321) one finds the expressions "ferric chloride" and "sodium chlorid," De Forcrand's definition of phenates and phenoxides, as having respectively the formulæ C_6H_5OH,MOH and C_6H_5OM, where M is a metallic atom is given on p. 294; but on p. 299 it is stated that from ammonium tribromophenoxide, $NH_4.O.C_6H_2Br_3$, other tribromophenates may be obtained. On p. 300 reference is made to ammonium tribromophenate and sodium tribromocresolate, and on p. 333 one finds three isomeric substances described as "orthocresoxide," "paracresoxide," and "metacresylate." These vagaries in nomenclature are perplexing to students who may consult this work.

The section on aromatic acids is a useful compilation which includes description of the chemical properties and analytical reactions of the principal aromatic carboxylic and sulphonic acids, together with fairly complete references to such important compounds as benzaldehyde (oil of bitter almonds), saccharin, cinnamaldehyde, coumarin, and vanillin.

In view of the interest attaching to salicylic acid as a preservative and a drug, considerable space is devoted to the detection and estimation of this substance in a large variety of commercial products. In this connection reference is made to the colorimetric methods of estimating salicylic acid, which were formerly described in the ANALYST (1903, **28**, 2; 1905, **30**, 124, and 1907, **32**, 286).

The analytical reactions of pyrogallol are summarised and compared with those of gallotannic and gallic acids in a special section devoted to these polyhydric phenols and their esters and ethers.

The section on phthalic acid and the phthaleïns contains a short account of the employment of phenolphthaleïn and similar indicators in acidimetry and alkalimetry.

G. T. MORGAN.

Modern Explosives.—Mr. A. Marshall's article on "Modern Explosives" occupies fifty-eight pages in the volume under review. The author had an opportunity, of which he has, unfortunately, not availed himself, to contribute a detailed and critical account of the methods of analysis of high explosives and smokeless sporting powders.

There is, in this respect, a distinct gap in the literature of explosives, and it is to be regretted that some effort was not made to deal with this branch of analytical chemistry. It is improbable that the analyst who is not connected with explosive manufacture would, in the course of general practice, have to report on cordite, guncotton, or nitro-glycerin; but, if this were the case, full information would be found in the Government specifications. On the other hand, it is quite possible that samples of either high explosives or sporting smokeless powders would be submitted to him. Should he turn to Mr. Marshall's article for help, no adequate information will be found. Even a tabulated list of the main groups of high explosives and sporting powders, with examples of the compositions of the more characteristic members of each group, would have been of use and assistance. In a work on commercial analysis, to confine attention, from the analytical point of view, almost wholly to the question of cordite and its ingredients is, in the reviewer's opinion, an error of some magnitude. Cellulose nitrates have been dealt with concisely, and the necessary references to recent literature are included. The writer cannot, however, from his own experience, agree with the author's statement that the Schultze-Tiemann method for the determination of nitrogen in guncotton gives results 0·1 per cent. to 0·2 per cent. higher than the Lunge nitrometer. On averaging a large number of comparative tests carried out by the same operator, the results are practically identical. The statement that all but the very low nitrated products of nitrocellulose are insoluble in pure alcohol is inaccurate. Collodion cotton, containing from 12·0 to 12·3 per cent. of nitrogen, intended for the manufacture of blasting gelatine, cannot be classed as one of the lowest nitrates; yet a good collodion cotton, intended for this purpose, will have a solubility, in absolute alcohol, of nearly 50 per cent. Whilst on this subject it should be mentioned that the author's suggestion that the easiest method of determining the suitability of collodion cotton for blasting gelatine is to investigate its viscosity in solvents, has only an academic interest. The only reliable laboratory test is to make a "jelly" with nitroglycerin containing 2 to 3 per cent. of collodion cotton by heating the mixture in a small porcelain basin on the water-bath. The section upon nitroglycerin also includes notes upon dinitroglycerin and the mixed esters of glycerol, and here it should be noted that on a single page four minus signs have been omitted in the data for freezing-points. Picric acid has been dealt with at a length in excess of the requirements of explosives, but, as tinctorial chemistry had also to be considered, it was perhaps natural that this part of the article should have been expanded to deal with a number of tests for detecting traces of picric acid. In dealing with fulminate of mercury, the author has very wisely quoted, at considerable length, from Brownsdon's work upon the analysis of cap composition. Under the heading of "Stability Tests" the well-worn paths of the Home Office Specification have been trodden once more, and the only point of interest here is that details of the heat-tests in use in the United States have also been included. The tests proposed by Guttmann, Spica, Vieille, Will, Bergmann, and Junk, and the German tests at 135° C., are shortly dealt with, and, in addition, the British Government silvered vessel test and the United States Army tests at 115° C. are briefly referred to. The question of the detection of mercury in explosives has been dealt with by quoting verbatim Messrs. Dupré's methods from the Annual Report of the Inspectors of Explosives for 1907, and the microscopic test of Hargreaves and Rowe is also referred to. Among instances of uncorrected errors, the following may be mentioned: Wood pulp and starch are stated to contain no oxygen. On p. 593 "saturated lime" should read "saturated brine," and the maximum percentage of moisture allowed in M.D. cordite should be 1·5 per cent., and not 1 per cent. On p. 615 the word "boiling" should be "heating."

GEORGE W. MACDONALD.

MICRO-ORGANISMS AND FERMENTATION. By ALFRED JÖRGENSEN. Translated by S. H. DAVIES, M.Sc. 1911. London: Charles Griffin and Co. Price 15s. net.

This new edition of Jörgensen's well-known textbook is the fourth which has appeared in English, and is a translation of the fifth German edition published in 1909. The wide extent of the investigations made during the course of the last ten years has necessitated considerable increase in the bulk of the book, which may, indeed, be regarded more as a new work than as a new edition.

The familiar characters of the previous issues are, however, retained, special attention being paid to the use of pure cultures of yeasts inaugurated by Hansen nearly thirty years ago. The work of the Danish school of micro-biologists is naturally accorded an important place, but that of other investigators also receives notice, although the discussion of the theory of fermentation contains no reference to the excellent work of Slator and only brief notices of the researches of Ehrlich and of Harden and Young.

The author first gives a short account of the methods employed in the microscopical and physiological examination of micro-organisms, the subjects treated of including staining, sterilisation, antiseptics, nutrient media, pure cultures, etc. This section might have been greatly increased in value by the inclusion of a more detailed description of the technique adopted in the author's laboratory. Next comes a discussion of the procedure followed in the biological examination of air and water, principally with reference to the requirements of the brewery. Chapter III. deals with the actions and conditions of growth of zymogenic bacteria, and Chapter IV. with the occurrence and life-history of most of the common mould-fungi.

The fifth section, occupying nearly one-half of the whole volume, is devoted to the yeasts, all the more important culture and wild varieties being considered in their relations to the fermentation industries. This part of the book gives, in addition, accounts of the history of fermentation and of the various investigations which have been carried out on variations in the *Saccharomycetes*, the morphology and anatomy of yeast-cells, and a number of allied matters. A description is also given of the manner in which Hansen applied the phenomenon of spore-formation to the analysis of yeasts.

Chapter VI., which has been considerably reduced in volume, deals briefly with the means employed for the preparation of mass-cultures of pure yeast for industrial purposes.

The book will commend itself to all those who, engaged in practical brewing and distilling, do not desire to carry on their work in a mere rule-of-thumb manner. The ground covered by the author is wide, and many of the questions dealt with are necessarily touched upon but lightly. But, even where this is the case, the brewer or distiller will be able to extract much valuable information and many suggestive hints on practical points.

The quality of the translation is, on the whole, good; the grammar and nomenclature are, it is true, faulty in places, but in no instance is the meaning obscure. Very few misprints are apparent.

A feature which increases the usefulness of the book, and which was not included in any of the previous editions, is the provision of a subject-index. A fairly complete bibliography is also given. T. H. POPE.

PHYSICO-CHEMICAL TABLES. Vol. II. By JOHN CASTELL-EVANS. London: Charles Griffin and Co., Ltd. 1911. Pp. xiv + 688. Price 36s. net.

This volume completes the work, and contains Part III., devoted to Physics, and Part IV., on Analytical Chemistry. The last section does not appear to be quite complete, as a number of tables which have appeared in current literature during the past few years, and which are of use to analysts, have not been included. Criticism on this point is disarmed by the fact that the work is a posthumous one.

In the publishers' note the work is truly described "as a fitting monument to the energy, perseverance, and deep devotion of the author," and no one studying the book can fail to be struck by the thoroughness and intimate grasp of the subject displayed. It might be urged as a possible failing that the source of the tables is not given, but on reading carefully it becomes apparent that no table is simply copied; all have been carefully checked, and in very many, if not in all, cases recalculated. The authority for the figures is John Castell-Evans. The very tables themselves inspire confidence, and one instinctively comes to feel that the omission of the source does not matter and that the guarantee of the author is sufficient.

The chief fault to be found with the work is also its greatest merit—its extreme accuracy. In very many cases, in a busy laboratory, a table is used to calculate results obtained under conditions which do not permit of more than three or four significant figures being reliable; Castell-Evans presents tables giving six, seven, or even nine figures, and their very bulk defeats the author's object. The book is for the reference library, or for use when the utmost precautions to insure accuracy are used, but not one to lie upon the working bench for daily use.

To review the Analytical portion seriatim—the tables of factors to convert weights of precipitates and c.c. of standard solution used into the constituent to be determined are unusually complete, but in some cases the factors are based on methods which are obsolete. Thus factors for volumetric boron estimation are given only for the method of titration with baryta till the solution is just clear of the turbidity which forms at first, while the method of titration in the presence of polyhydric alcohols, which requires a factor just half that given, is ignored. For glycerol, Hehner's bichromate method and Benedikt's acetin method are not mentioned, and none of the factors given, which are for methods now superseded by the above, apply. No other table requires comment till the section of Alcoholimetry is reached. This is a marvellous compilation, nearly complete, and a masterly review of the work which has been done, but all mention of proof spirit has been omitted. Possibly this was intentional, and certainly most analysts will agree that "proof spirit" is a relic of the barbarous past, but so long as it is a legal expression of alcoholic strength it should appear in alcohol tables.

Much of the Physical portion will be of use to the analyst. Over 100 pages are devoted to specific gravities of solutions of almost every substance of usual occurrence, and such subjects as solubilities, viscosity, and surface tension are treated of in a useful manner. The present volume, like its predecessor, is one which will well repay, not mere perusal, but careful study by the analyst, as it will not only give him an insight into those branches of science of which more use might be made in analytical chemistry, but will show him where he can turn in case of need.

H. Droop Richmond.

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INSTITUTE OF CHEMISTRY.

The Thirty-third Annual General Meeting of the Institute of Chemistry was held at 30, Bloomsbury Square, on March 1, Dr. George Beilby, F.R.S., President, in the chair.

After the routine business had been transacted, the President delivered his address. After referring to the losses of the Institute by death, mentioning especially Professor Campbell Brown, Mr. Michael Carteighe, and Mr. Oscar Guttmann, he dealt with the progress and work of the Institute. The membership continued steadily to increase, notwithstanding the high standard of the examinations; while the position of the Institute, as an organisation existing for public service, never stood so high as at the present time. Over 96 per cent. of the appointments of public analysts in the United Kingdom are held by Fellows of the Institute, the Final Examination in the Chemistry of Food and Drugs being conducted on lines prescribed by the Local Government Board, and specially recognised by them for these appointments.

The Council had under consideration the institution of a series of lectures, the object of which would be to bridge over the gap between academic training and practice. Without depreciating the value of a broad scientific education, it was realised that students entirely trained in an academic atmosphere missed some of the advantages of the old system of private pupilage in the laboratories of practising Fellows or in works. This loss was met, in some instances, by students preparing for the Final Examination in such laboratories; but there was a need for a means of introducing something analogous to the clinical instruction afforded to medical students. It was proposed, therefore, to ask Fellows having expert knowledge to give lectures which would give students an insight into the actual work of the chemist, whether engaged in the application of his science to commerce and industry, or to the scientific control of the affairs of daily life. Apart from the value of the information which could be imparted by such experts, considerable advantage would accrue to the students from the mental contact with such men, while they would acquire a proper sense of proportion in respect of the work of chemists in various branches of practice.

The President then remarked on the successful working of the Appointments Register, by which means the Institute introduced properly qualified chemists to authorities and manufacturers requiring their services, and he acknowledged the valuable help given by the Honorary Corresponding Secretaries of the Institute to members taking up appointments in various parts of the Empire.

Continuing, he referred to the appeal which had been made for funds for new buildings. It had been estimated that £15,000 would be required for this purpose, and the Council were able to record promises amounting to £7,566, of which about £5,000 had actually been received. This included contributions from four of the great city companies and from a few manufacturing firms. He hoped that others would show their appreciation of the work of the Institute in the same practical manner, and he appealed to the Fellows and Associates themselves, as far as they were able, to promote the successful raising of the fund.

Professor MELDOLA, in moving a vote of thanks to the President for his address, said it would give him much pleasure to see the Institute take an active part in the education of chemists, and he cordially supported the new departure as to the institution of the proposed lectures, which would make the student realise more fully the value of the practical side of his subject. The Institute had always been happy in the selection of its officers, and he (Professor Meldola) paid a high tribute to the services of Dr. Beilby, the present President, for his zeal and attention to the affairs of the Institute.

The following Fellows were re-elected as Censors: Professor Percy F. Frankland, Mr. David Howard, Professor J. Millar Thomson, and Sir William Tilden.

The following were elected Officers and Council:

President.—George Thomas Beilby, LL.D., F.R.S.

Vice-Presidents.—Frank Clowes, D.Sc.; Percy Faraday Frankland, LL.D., Ph.D., F.R.S.; George McGowan, Ph.D.; Raphael Meldola, F.R.S.; John Millar Thomson, LL.D., F.R.S.; Sir William Augustus Tilden, D.Sc., F.R.S.

Hon. Treasurer.—Alfred Gordon Salamon, A.R.S.M.

Members of Council.—Francis William Frederick Arnaud; Edward John Bevan; Bertram Blount; William John Atkinson Butterfield, M.A.; Francis Howard Carr; Charles Edward Cassal; Alfred Chaston Chapman;* Arthur Crozier Claudet, A.R.S.M.; James Connah, B.Sc.; William Salvador Curphey; Cyril Dickinson, B.Sc.; James Johnston Dobbie, Ph.D., LL.D., F.R.S.; William Porter Dreaper; John Alfred Foster; Walter Charles Hancock, B.A.; Frank William Harbord, A.R.S.M.; Frederick Gowland Hopkins, M.A., M.B., D.Sc., F.R.S.; William Jackson Pope, M.A., M.Sc., F.R.S.; Thomas Slater Price, D.Sc., Ph.D.; Charles Proctor; Sir Boverton Redwood, D.Sc.; Henry Droop Richmond; Alfred Smetham; Edward William Voelcker, A.R.S.M.: John White; Sydney Young, D.Sc., F.R.S.; William Maurice Gathorne Young.

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* Mr. Chapman, who has been appointed examiner, has resigned his seat on the Council, and Sir Alexander Pedler, C.I.E., F.R.S., has been co-opted in his place.

MAY, 1911. Vol. XXXVI., No. 422.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The ordinary meeting of the Society was held on Wednesday evening, April 5, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. W. Voelcker, A.R.S.M., F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. H. G. Battye, G. D. Elsdon, B.Sc., N. Evers, B.Sc., A.I.C., and A. W. Knapp, B.Sc., F.I.C., were read for the second time; and a certificate in favour of Mr. Alfred Douglas Heywood, A.I.C., 73, High Street, Southend-on-Sea, chemist to the Hygienic Dairy Society, Salisbury, was read for the first time.

Messrs. J. Clifford, A.R.C.S.I., C. H. La Wall, J. Connah, B.Sc., F.I.C., H. F. Knight, W. R. Mummery, F.I.C., and W. M. Seaber, B.Sc., F.I.C., were elected members of the Society.

The following papers were read: "The Analytical and Microscopical Examination of Compound Liquorice Powder," by G. E. Scott-Smith, F.I.C., and John Evans, F.I.C.; "Note on Gerber's 'Neusal' Milk Test" and "Note on Abnormal Cotton Cakes," by John Golding, F.I.C.; "A Further Contribution to the Question of Turpentine Substitutes," by J. H. Coste, F.I.C., and L. Myddelton Nash, F.I.C.; "Note on Almond and Apricot Kernel Oils" and "Constants of Chicken and Turkey Fats," by Raymond Ross, F.I.C., and Joseph Race, F.I.C.

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EXAMINATION OF SHREWSBURY AND KNAPP'S PROCESS FOR THE ESTIMATION OF COCOANUT OIL.

By RAYMOND ROSS, F.I.C., JOSEPH RACE, F.I.C., AND FRANK MAUDSLEY, B.Sc., F.I.C.

(*Read at the Meeting, March* 1, 1911.)

Last year Messrs. Shrewsbury and Knapp described a process for the estimation of cocoanut oil in butter (Analyst, 1910, **35**, 385). Although a considerable number of English butters were examined, very few of foreign origin were investigated, and of the three examples given in this latter division two were classed as suspicious.

It therefore appeared advisable to examine a number of foreign butters and cocoanut oils by this process. The samples in question were obtained for us from the wholesale market, and every possible precaution was taken to insure their authenticity. The following results were obtained :

Butter.	Shrewsbury and Knapp Figure.	Iodine Value.	Per Cent. of Triolein (calculated).	Shrewsbury and Knapp Figure of Residual Fats calculated at Triolein = 28.	Shrewsbury and Knapp Figure of Residual Fats calculated as Triolein = 65.
Finnish	31·5	41·3	47·9	18·1	0·4
Danish	30·0	41·5	48·0	16·5	−1·2
Siberian	33·5	38·7	44·9	21·0	4·3
Swedish	33·0	—	—	—	—
Irish Creamery	32·0	34·7	40·2	20·8	6·0
Irish Factory	35·5	39·3	45·6	23·0	5·9
Irish Dairy	28·5	39·1	45·4	16·0	−1·0
St. Ivel (English)	30·5	37·5	43·5	18·0	2·2
Kiel	31·5	32·4	37·6	21·0	7·1
Cocoanut stearine	138·5	5·4	6·3	136·8	—
Cocoanut oil (Ceylon)	170·5	5·8	6·7	168·6	—
,, ,, (Epicos)	165·5	6·5	7·5	163·4	—
,, ,, (Cochin)	166·5	6·6	7·6	164·1	—
,, ,, (Colombo)	168·5	6·3	7·3	166·5	—
Cocoa butter	16·5	31·2	36·2	6·4	—

Shrewsbury and Knapp (*loc. cit.*) give 32 as a suggested maximum, but our results seem to point to a maximum of 35·5.

The effect of variations of the amounts of triolein in butter is dwelt on by these authors. Our experience with triolein is distinctly unsatisfactory, as great variations in the number obtained occur. This is probably due to the fact that a really good separation of the fatty acids in the alcohol-water solution rarely takes place. With oleic acid, however, fairly uniform results were obtained, the figure varying from 25 to 29·5 with the various samples at our disposal.

The figure 65 for triolein, and more so the somewhat higher ones obtained by us, must be excessive, because butters which differ considerably in their percentage of triolein as measured by the iodine value do not differ proportionately in their Shrewsbury and Knapp figure. This would not be likely if 65 or any higher figure were correct for triolein. Probably the number obtained by us for oleic acid is approximately right.

In the table given above the percentage of triolein is calculated from the iodine absorption, and from this the Shrewsbury and Knapp number is obtained by calculation for the residual fats on the assumption of a Shrewsbury and Knapp figure for triolein of 28 for one column and 65, as given in their paper, for the other. From these it is evident that, if 65 were correct, no value could be assigned to anything but the amount of oleic acid present, and the whole process would be no better than an iodine value. That this is not the case is seen from the table, where a rise in iodine absorption does not necessarily imply a rise in the Shrewsbury and Knapp figure.

The constitution of the residual fatty acids after separation from the alcohol water solution is given below, and is rather interesting. It seems to point to proportional quantities of all the constituents being dissolved. The main factor in the Shrewsbury and Knapp figure would be dependent then, as contended by the authors, on the proportion of lower and more soluble fatty acids present:

RESIDUAL FATTY ACIDS.

Name.	Iodine Value.	Zeiss at 45° C.	Molecular Weight.	Oleic Acid Per Cent. (calculated).	M – O.
Finnish...	44·0	31·5	259·7	49·0	238·2
Danish	47·0	31·5	259·1	52·2	234·1
Siberian	40·2	31·0	263·3	44·7	248·3
Irish Creamery ...	39·5	30·5	264·0	44·0	250·0
Irish Factory	40·8	31·5	257·0	45·3	236·4
Irish Dairy	43·2	32·0	261·3	48·0	242·3
Swedish	—	31·0	—	—	—

In considering the genuineness or otherwise of a butter, it is doubtless advisable not to rely entirely on any particular figure, but to consider the case as a whole. A figure that we have found to give instructive results is obtained by calculating the iodine value of the fatty acids to oleic acid, and from this the molecular weight of the residual fatty acids, by the following formulæ:

$$\frac{\text{Iodine value per cent.} \times 100}{90} = \text{oleic acid} = \text{O},$$

$$\text{Molecular weight of residual fatty acids} = \frac{100\ \text{M} - (282 \times \text{O})}{100 - \text{O}},$$

where M = molecular weight of the fatty acids and O = oleic acid per cent. The figure thus obtained for genuine butters is generally about 235, and the addition of cocoanut oil tends to reduce the figure considerably.

We find that it is absolutely necessary to follow the process exactly as described by Shrewsbury and Knapp, especially as regards glycerol saponification. With alcoholic potash only extraordinarily high results were obtained; for example, Swedish butter gave 112, and Danish butter 77. If, however, the correct quantity of glycerol is added after saponification, the results are normal.

The method will probably be of use in helping to determine the approximate composition of margarine. Thus a high-class margarine, A, showed very different results from a cheap one, B, and the former probably contained a considerable amount of cocoanut oil:

	A.	B.
Zeiss butyro-refractometer (25° C.)	52·5	61·0
Sp. gr. (37·8° C.)	0·9097	—
Shrewsbury and Knapp figure	47·5	19·5
Koettstorfer value	232·8	204·9
Per cent. of insoluble acids left in funnel after Shrewsbury and Knapp	65·8	85·1
Reichert-Meissl	9·0	4·7
Polenske	6·7	1·4

Mr. Knapp sends the following note:

"The figure given by us for triolein was obtained from a single sample, which had a butyro-refractometer reading at 40° C. of 59·0. Lewkowitsch gives 56·5 for pure triolein. I think it would add to the value of the several figures obtained by Messrs. Ross, Race, and Maudsley for oleic acid if they indicated the purity of their samples. With regard to their calculation to show that 65 is too high a figure for triolein, it should be remembered that this calculation is based on the supposition that the various fatty acids do not interfere with each other's solubility. In the process, we are actually dealing with the relative solubilities of oleic, etc., acids in the water-alcohol mixture and in the separated fatty acids. What is required is the figure which olein gives in presence of the other butter constituents, and the calculation very usefully shows that 65 is too high for this figure. Mr. Shrewsbury and I would welcome 28 as the figure for olein, as it would increase the value of the process, and enable us to understand why both cocoanut-oil-free margarine and lard give lower Shrewsbury and Knapp figures than butter."

* * * * *

THE ANALYTICAL AND MICROSCOPICAL EXAMINATION OF COMPOUND LIQUORICE POWDER.

By G. E. SCOTT-SMITH, F.I.C., and JOHN EVANS, F.I.C.

(*Read at the Meeting, April* 5, 1911.)

COMPOUND liquorice powder, prepared in accordance with the directions of the British Pharmacopœia, contains:

	Per Cent.
Senna, in fine powder	16·7
Liquorice root, in fine powder	16·7
Fennel fruit, in fine powder	8·3
Sublimed sulphur	8·3
Refined sugar, in powder	50·0
	100·0

We understand that it is customary on the manufacturing scale to grind the whole ingredients into powder together, gradually sifting and returning the gruffs to the mill until they are reduced to a very low bulk, usually a few pounds. These are set aside and added to a succeeding batch, which is then treated in the same manner. By this process the correct ratio of the various ingredients is slightly altered, as the sulphur and sugar yield no gruffs, while the senna, liquorice, and fennel yield gruffs in varying quantities.

During the last three years we have examined over 100 samples of compound liquorice powder, the majority of which were submitted by the Inspectors under the Sale of Food and Drugs Acts. We have been much impressed with the great diversity in appearance of the samples, and several instances of careless manufacture have come under our notice. In several samples the sulphur was present to the extent of 16 per cent.—that is, nearly double the quantity prescribed by the British Pharmacopœia—whilst in other samples the sulphur was entirely omitted. Again, in our microscopical examination of several samples, the liquorice root was so predominant that the senna and fennel were identified with difficulty, and might justly be described as traces.

Many instances of gross adulteration with ground olive stones and ground almond shells have also come under our notice. These adulterants, we understand, find their way into compound liquorice powder through using foreign ground liquorice root, which is subject to these adulterations.

Upon referring to the literature on the subject, we find that the following standards have been suggested:

1. The Local Government Board (Ireland): Ash, 4·5 per cent.

2. J. C. McWalter (*Pharm. J.*, 1902, **69**, 89): Moisture should be from 4 to 6 per cent.; ash, not above 5 per cent.; soluble ash, not exceeding 3 per cent. Not less than 4 per cent. should be dissolved in 70 per cent. alcohol.

3. C. G. Moor ("Suggested Standards for Food and Drugs," p. 179): Water should not exceed 6 per cent.; ash should be about 4 to 5 per cent.; soluble ash about 2·5 per cent.

4. H. W. Gadd (*Year-Book of Pharmacy*, 1901, p. 390) gives the following results of analysis of three samples of compound liquorice powder:

	Moisture per Cent.	Ash per Cent.	Soluble Ash per Cent.	Extractive in Tincture per Cent.*
A	6·35	4·5	2·80	4·83
B	5·40	4·5	3·15	3·73
C	4·15	4·8	2·68	4·47

5. J. F. Liverseege (*Year-Book of Pharmacy*, 1906, p. 269): The amount of ash found in twenty-one samples was 3·8 to 6·4 per cent. This author suggests a method

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* The tincture was prepared by macerating 5 grms. of the powder in 70 c.c. of 70 per cent. alcohol.

of analysis by extracting the powder first with strong alcohol; then with carbon bisulphide, to obtain the sulphur; and finally with water, to obtain the sugar.

The amount of moisture in the samples examined by us varied from 3·7 to 4·2 per cent.

The following table shows the amount of ash found in commercial samples:

	Maximum.	Minimum.	Average.
Total ash	6·78	4·36	5·35
Insoluble ash	3·98	1·62	2·54
Soluble ash...	3·20	2·48	2·80

Several samples not included in the above table contained abnormal proportions of total ash, even amounting to 8·5 per cent. In all these samples the ash insoluble in hydrochloric acid (sand) was excessive, amounting to nearly 4 per cent. In our opinion these samples were prepared from inferior or improperly cleaned drugs.

The disturbing action of the sulphur on the total and insoluble ash, due to the combination of the sulphur with the mineral constituents of senna, liquorice, and fennel, was pointed out by one of us in a previous paper (*Pharm. J.*, 1905, **74**, 363).

The presence of sulphur in the preparation not only causes an increase of total ash over the actual amount of mineral matter present in the several ingredients, but it also converts some of the insoluble into soluble ash.

The following table will illustrate this point:

	Compound Liquorice Powder. B. P.	Compound Liquorice Powder without Sulphur + 8 per Cent. Extra Sugar.
Total ash	4·66	3·75
Insoluble ash	1·68	2·76
Soluble ash	2·98	0·99

These two samples were prepared from the same ingredients.

Thus it will be seen that the percentage of soluble ash is of no value in detecting exhausted drugs in compound liquorice powder; even if exhausted drugs had been employed, the percentage of soluble ash would still be raised, due to the conversion of the insoluble carbonate into soluble sulphides and sulphates. Again, the amount of extractive matter yielded to 70 per cent. alcohol, as suggested by some observers, is of little value for detecting the presence of exhausted ingredients, as the whole of the sugar is soluble in 70 per cent. alcohol; thus the alcoholic extract would contain a large amount of sugar, together with a smaller amount of extractive matter from the drugs, and a slight variation in the proportion of sugar would vitiate the results. Sugar is soluble even in 95 per cent. alcohol.

The determination of the sulphur presents no special difficulty. The following is the method employed by us: One grm. of the powder is treated with 15 to 20 c.c. of concentrated nitric acid and heated; and if the sulphur fuses, a small quantity of bromine is added. When oxidation is complete, hydrochloric acid is added, and evaporated to remove excess of nitric acid, diluted with water, and filtered from any siliceous matter, and finally precipitated as barium sulphate. The amount of sulphur actually present, either as sulphates or organic sulphur, in the senna, liquorice, and fennel, is very small, and may be neglected. The determination of the sulphur by means of carbon bisulphide cannot, in our experience, be relied on, as commercial samples of sublimed sulphur are not always completely soluble in this reagent.

Before entering upon the consideration of the adulteration of compound liquorice powder with foreign vegetable powders, it is necessary that we should be thoroughly acquainted with the microscopic characters of those ingredients of which it is composed. The media employed by us in the routine microscopical examination of compound liquorice powder are water, glycerine, and a strong solution of chloral hydrate (50 grms. in 20 c.c. of water). Iodine water is also used for the better observation of the starch granules.

The histology of the senna leaf presents many characters by which it can be easily identified, even in such a complex mixture as compound liquorice powder. It has an iso-bilateral structure—that is, there is palisade tissue on both the upper and under surfaces—and between the two palisade tissues there is a layer of spongy parenchyma. It has peculiar hairs scattered over both the lower and upper surfaces. These hairs are one-celled and conical, they may be straight or curved, and are usually slightly contracted at the base. They are thick-walled and have a warty appearance. The stomata are surrounded by two large cells, which are parallel to the ostiole or opening of the stomata, and as a rule one of these cells is much larger than the other. The epidermal tissue is further characterised by the scars of fallen hairs and the peculiar radiating arrangement of the cells surrounding the base of the hair. Besides the foregoing, elements derived from the bundles of the midrib and veins possess certain characteristics. Thus, in a longitudinal section of a leaf bundle, wood vessels are followed by pericylic fibres, and outside these comes a single layer of parenchymatous cells containing prismatic crystals of calcium oxalate. Though the presence of these crystal cells is not strictly diagnostic, a strip of the senna leaf bundle can always be recognised in a microscopic preparation of powdered senna by the sequence of the elements—namely, wood vessels, pericylic fibres, and a single row of cells containing prismatic crystals.

The British Pharmacopœia describes liquorice root as the peeled root and peeled subterranean stem of Glycyrrhiza glabra. It is this decorticated portion that should be used in the manufacture of galenical preparations. It contains about 30 per cent. of starch, and the predominating feature of powdered liquorice root, when examined microscopically, is the abundance of starch granules. These granules vary both in outline and size, and present no distinct hilum or markings. They vary in outline from circular to elongated ellipses, but the majority are circular or elliptical. In size they vary from mere dots to even 20μ, and they average about 10μ. Some of the granules have even slight protuberances resembling budding yeast cells. Liquorice root is further characterised by groups of thick-walled yellowish fibres and large thick-walled yellow-pitted vessels. The yellow colouring matter of liquorice root is almost entirely situated in the fibres and vessels. The best fibres of liquorice root are accompanied by regular rows of cells containing single prismatic crystals of calcium oxalate.

Fennel is the dried ripe fruit of *Fœniculum capillaceum* (N.O. Umbelliferæ), and consists of two readily separated mericarps. Each mericarp has five primary ridges, and in a transverse section shows six large vittæ, or oil canals. In the powder, fragments of the vittæ are easily recognised by their dark brown colour. Powdered fennel may be identified by the presence of large parenchymatous cells with spiral or reticulate thickening. The cells of the endosperm are polygonal in shape and have rather thick walls, and contains aleurone grains, oil globules, etc. Starch is absent. Groups of narrow elongated cells, arranged parallel to one another, are also seen in the powder; they are derived from the inner epidermis of the fruit. The fragments of vessels, sclerenchymatous fibres and tracheids, are derived from the fibro-vascular bundles of the fruit, and from the carpophore and pedicel of the fruit.

Mention was made earlier that some of the adulterants we had met with consisted of ground olive stones and ground almond shells. Fig. IV. is a characteristic example of compound liquorice powder adulterated with ground olive stones. The elements of senna, liquorice, and fennel are shown, and in addition will be observed peculiar sclerenchymatous cells which are foreign to any of the constituents of compound liquorice powder, and characteristic of such things as olive stones, date stones, and almond shells.

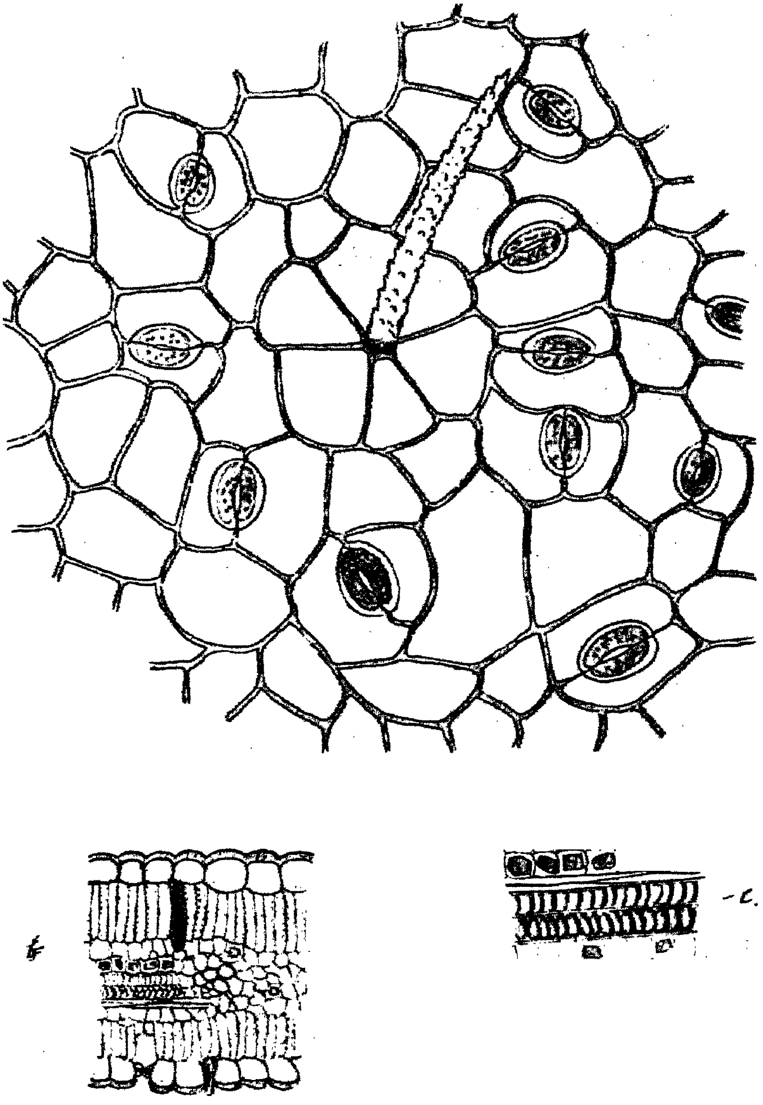
DISCUSSION.

Mr. C. A. Hill said that the ash of powdered Alexandrian senna was higher by 1 or 1½ per cent. than that of Tinnevelly senna. In a sample of compound liquorice powder which he had examined for a special purpose, the ash, calculated from the ash contents of the several constituents, worked out at 3·79 per cent., while the compound powder yielded 4·25 per cent. of ash. The question of purity or genuineness was only likely to arise with the cheapest qualities of compound liquorice powder, which would certainly be made with Tinnevelly senna.

Mr. F. W. Richardson said that he had not found the proportion of ash to be of any great assistance, but it was very useful to examine microscopically the fibre obtained after treatment with dilute acid and alkali. This fibre generally amounted to about 16 per cent. The proportion of matter soluble in water, apart from the sugar, was usually about 12 per cent., and a determination of the total soluble matter, which with the sugar should amount to about 62 per cent., was also very useful. It might be mentioned that the proportion of sulphur even in genuine samples was liable to considerable variation, since the powder was sometimes comparatively coarse, and separation of the ingredients occurred.

* * * * *

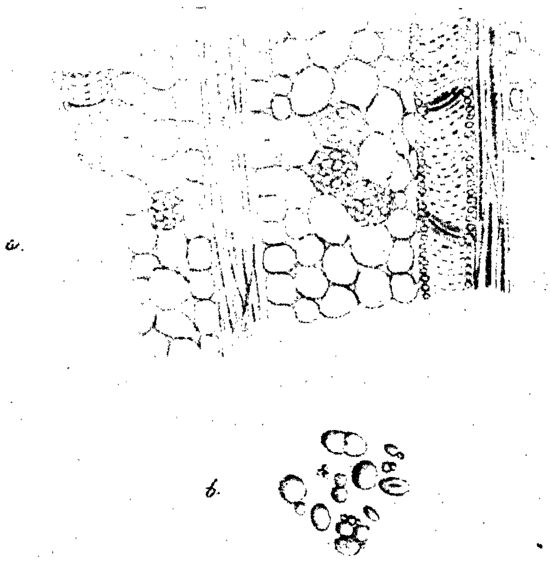
FIG I.



a, EPIDERMIS OF SENNA LEAF. *b*, TRANSVERSE SECTION OF SENNA LEAF.
c, LONGITUDINAL SECTION OF MIDRIB.

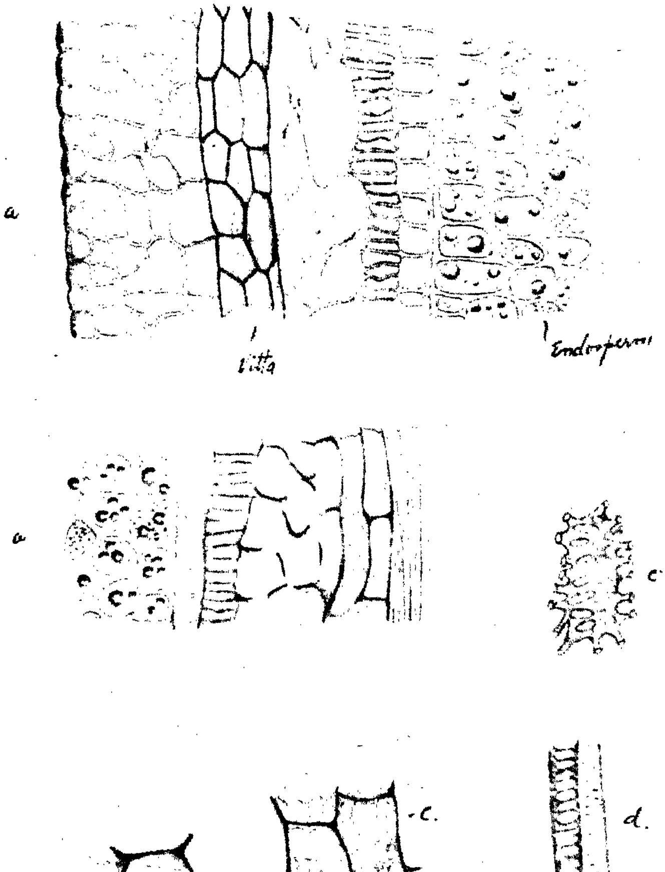
[*To face page* 202.

FIG. II.



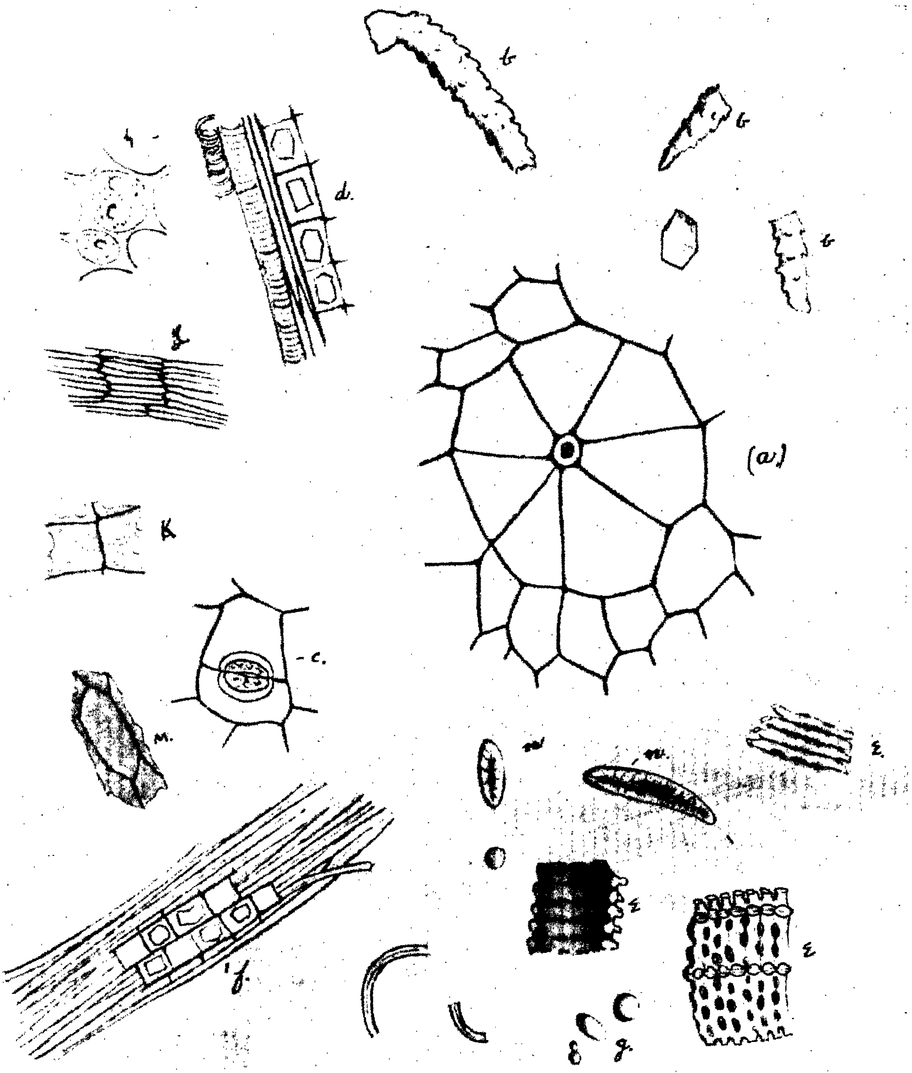
a, LONGITUDINAL SECTION OF LIQUORICE ROOT.
b, STARCH GRANULES.

Fig. III.



a, Longitudinal Section of Fennel. *c* Thickened Cells of Mesocarp.
b, Inner Epidermis of Pericarp. *d*. Portions of Pedicel of Fruit.

FIG IV



a, EPIDERMIS OF SENNA LEAF.
b, HAIRS AND FRAGMENTS OF HAIRS OF SENNA.
c, PORTION OF EPIDERMIS WITH STOMA.
d, FRAGMENT OF FIBRO-VASCULAR BUNDLE OF SENNA.
e, PORTIONS OF VESSELS OF LIQUORICE ROOT.
f, SCLERENCHYMATOUS FIBRES OF LIQUORICE ROOT.
g, STARCH GRANULES OF LIQUORICE ROOT.
h, ENDOSPERM OF FENNEL FRUIT.
k, CELLS OF MESOCARP OF FENNEL.
l, INNER EPIDERMIS OF PERICARP OF FENNEL.
m, PORTION OF VITTA, OR OIL CANAL.
n, ISOLATED STONE CELLS FOREIGN TO LIQUORICE POWDER.

THE "NEUSAL" MILK TEST.

By J. GOLDING, F.I.C.

(*Read at the Meeting, April 5,* 1911.)

The difficulties which the "Neusal" milk test (invented by O. Wendler) is designed to overcome do not exist for the analyst working in his own laboratory. Certain points of chemical interest are, however, brought out by an investigation of the method, and also the greatly-extended use to which the mechanical testing of milk is now put, frequently involves the calling in of expert advice on the methods chosen and the results obtained.

It is only necessary to mention, as an example, the valuable work done by the Milk Record Societies, where men are employed to travel round a circuit of farms testing and weighing the milk from each cow in the herd both night and morning, to give an instance of a case where great advantage would accrue were it possible to replace the large quantities of sulphuric acid required for Gerber's acid test by some more portable reagent. To meet this difficulty various methods have been tried, such as the "Sin-acid" method, and Gerber's "Sal" method. In these, solutions of alkaline mixtures of salts were used with amyl or butyl alcohol (added separately), to dissolve the milk.

In 1905 I investigated these methods, and came to the conclusion that both methods were unreliable, because, when the milk is dissolved with an alkaline solution, the fat which separates consists not only of butter-fat, as in Gerber's acid method, but that the volume is swollen by the alcohol which the fat dissolves from these alkaline solutions of the milk. For this reason slight inaccuracies which could hardly be avoided in measuring the alcohol resulted in considerable variations in the volume of fat to be read off; also, there was nothing to show that this had taken place. Allowance is made in Gerber's "Sal" method by taking 10 c.c. of the milk in the same bottles in which 11 c.c. were required for the acid test. Variations of 0·5 to 1·5 c.c. of the amyl alcohol made no difference in the acid test, but in the "Sal" test such variations caused the fat layer to be more than doubled. It is, therefore, not surprising that these methods have not replaced the older acid methods.

The patent of Dr. O. Wendler for the "Neusal" method states that the milk is treated with a mixture consisting of one or more salts of a hydroxybenzoic acid (*e.g.*, sodium salicylate), a sucrate, and a fat-clarifying agent (butyl alcohol); the quantity of fat separating from the milk is then measured. The salt is sent out in a bag containing 260 grms., which is directed to be dissolved in 600 c.c. of water. The salt gives a blue coloured solution; with this is also a bottle containing 255 c.c. of isobutyl alcohol of a certain purity. This has to be added to the blue solution, and the bottle rinsed out with the liquid. The alcohol, though fairly insoluble in water, is very soluble in the solution of the salt. I have also found that a solution of sodium salicylate readily dissolves the alcohol with frothing, making a clear solution. The total volume of the solution obtained by dissolving the alcohol in the salt solution is about 1,000 c.c.

Two sizes of butyrometer tubes are supplied; to the smaller of these 2 c.c. of this "Neusal" solution are added, and 4·85 c.c. of milk. To the larger 4 c.c. of "Neusal" solution, and 9·7 c.c. of milk. The bottles are then corked, well shaken, and placed in water at 50° C. for four minutes, shaken again, and spun in the centrifugal machine for three minutes. It is then directed to place them in water at 45° C. for three minutes, after which the fat may be read off. If the acid butyrometers are to be used, the "Neusal" solution is diluted with an equal volume of water, and 12 c.c. of the mixture used with 9·7 c.c. of milk.

The sodium salicylate and butyl alcohol completely digest the milk at this temperature, and a very clear reading of the fat is obtained. Examples of the results obtained are given in the following table:

TABLE I.

Sulphuric Acid.	—	—	—	10 c.c.	Rose Gottlieb (*a*).
Milk.	4·85 c.c.	9·7 c.c.	9·7 c.c.	11 c.c.	Extraction (*b*).
"Neusal."	2 c.c.	4 c.c.	12 c.c. diluted.		
	Small Tubes.	Medium.	Large.	Acid.	
Milk, No. 1 ...	3·75	3·75	3·70	3·75	3·82 (*a*)
	3·85	3·80	3·70	3·75	3·82 (*b*)
" No. 2 ...	3·45	3·45	3·45	3·40	3·56 (*a*)
	3·40	3·45	3·45	3·45	3·54 (*b*)
" No. 3 ...	4·55	4·45	—	4·50	4·57 (*b*)
" No. 4 ...	1·45	—	—	—	1·53 (*b*)
" No. 5 ...	3·00	3·00	3·15	3·00	3·00 (*b*)
" No. 6 ...	—	—	6·00	6·00	6·09 (*b*)

I find that it is necessary to read the "Neusal" tubes to the bottom of the meniscus, while with the acid tubes I get results agreeing most nearly with the extraction method by reading to the middle of the meniscus. The "Neusal" tubes seem to require slightly longer spinning than the acid tubes; but if they are not spun sufficiently, a pearly appearance is seen at the bottom of the fatty layer, which serves as a valuable warning. Similar results are reported by M. Siegfeld in the *Molkerei-Zeitung* for May, 1910.

It is obvious from the fact that 9·7 c.c. of milk are taken in the same tubes in which 11 c c. are taken for the acid method, and in both the same volume of fat is read off that in this method as with the "Sal" method we are reading off a volume of fat plus dissolved alcohol; and, as would be expected, experiments in which varying quantities of "Neusal" were taken with the same quantity of milk showed this to be the case.

TABLE II.—ACID BUTYROMETER TUBES TAKEN WITH MILK CONTAINING 3·8 PER CENT. OF FAT.

"Neusal."	Water.	Milk.	Fat.
c.c.	c.c.	c.c.	Per Cent.
5·5	6·5	9·7	3·40
6·0	6·0	9·7	3·75
6·5	5·5	9·7	3·80
7·0	5·0	9·7	3·90
7·5	4·5	9·7	3·90

The experiments were repeated in tubes to which 0·5 c.c. of melted butter-fat had been added (see Table III.).

TABLE III.—0·5 C.C. BUTTER-FAT AT 50° C., TAKEN IN GERBER TUBES WITH—

Water.	Dilute "Neusal."	"Neusal" Alcohol.	50 per Cent. H_2SO_4.	1 per Cent. H_2SO_4.	1 per Cent. NaOH.	Fat.
21·5	—	—	—	—	—	3·60
9·5	12·0	—	—	—	—	4·50
15·5	6·0	—	—	—	—	3·70
—	21·5	—	—	—	—	5·35
19·5	—	2	—	—	—	4·50
—	—	2	19·5	—	—	3·60
—	—	2	—	19·5	—	4·50
—	—	2	—	—	19·5	4·50

TABLE IV.—EFFECT OF VARYING THE QUANTITY OF ALCOHOL: MILK OF 3 PER CENT.; FAT, 9·7 C.C.; 88 GRMS. OF SODIUM SALICYLATE MIXTURE DISSOLVED IN 200 C.C. OF WATER.

Solution.	Water.	Alcohol.	Result.
c.c.	c.c.	c.c.	
5	about 6	less than 1·4	cloudy fat
5	" 6	1·4	3 per cent. fat
5	" 6	1·5	3 per cent. fat, clear
5	" 6	1·6	3 per cent. fat
5	" 6	1·8	3·5 per cent. slightly milky
5	" 6	2·0	3·8 " " "
5	" 6	2·5	5 per cent. cloudy
5	" 5	3·0	about 6 per cent. cloudy

The variations are not so great as when more alkaline solutions are used (over 7 per cent. obtained). The table shows that the fat dissolves the alcohol from the solution. Varying the quantity of "Neusal" salt produced no alteration in the percentage of fat even if the quantity recommended was doubled; with less salt the readings of fat could not be obtained. In these experiments the alcohol was added separately (1·5 c.c.). The effect of varying the quantity of alcohol is shown in Table IV.

In conclusion, while the method gives satisfactory results when the exact quantity of alcohol is used, the fact that it is not pure fat which is being measured, but a solution, the volume of which will vary with the quantity of alcohol used, makes the method inferior in accuracy to the acid Gerber method, a fault which is likely to be increased if it is put into the hands of inexperienced persons. On the other hand, it is cheaper, safer, and more portable, and might be safely recommended to dairy farmers and others, to replace the acid method where this offers serious disadvantages; but the introduction of another possible error will prevent it from replacing the original Gerber method for more accurate work, though the method is a great advance on the previous alkaline methods.

DISCUSSION.

Mr. RICHMOND remarked that the fact that the method depended upon the differential solubility of iso-butyl alcohol in the fat and in the aqueous portion seemed to be a disadvantage. According to Mr. Golding's figures, the butyl alcohol appeared to be twice as soluble in the fat as in the aqueous portion, and it was obvious that such approximation to correctness as was obtained was due simply and solely to strict adherence to the conditions specified. This meant that one was entirely in the hands of the manufacturers, and, since the composition of the reagents in such cases was generally more or less secret, there was no means of ascertaining for oneself whether the correct conditions were really being fulfilled. The acid method was much more reliable, but here also, even in experienced hands, the results were liable to vary, as was instanced by the fact that, whereas Mr. Golding obtained correct results by reading half-way down the meniscus, he (Mr. Richmond) found it necessary to read, if anything, a little below the meniscus. Both Mr. Golding and himself knew what were the causes of these differences and could allow for them, but the unskilled person could not. As a matter of fact, all these methods—even the acid method—were merely approximate ones, and although in trained hands they might be satisfactory, the results were necessarily less trustworthy than those of actual analysis.

Mr. GOLDING said that the difference in the reading point was probably due to the fact that, while he used a hand-worked apparatus, Mr. Richmond had the advantage of mechanical power, and thus probably was able to extract the fat more completely.

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A FURTHER CONTRIBUTION TO THE QUESTION OF TURPENTINE SUBSTITUTES.

By J. H. COSTE, F.I.C., and L. MYDDELTON NASH, F.I.C.

(Read at the Meeting, April 5, 1911.)

There is abundant evidence of the presence of cyclic hydrocarbons (benzene and its homologues) in greater or less amount in petroleum from almost all sources.* In some varieties the proportion is very considerable. The examination by one of us of several materials sold as turpentine substitutes showed that these substances contained large percentages of such hydrocarbons, in addition to the more commonly recognised paraffins or cyclo-paraffins (Analyst, 1910, **35**, 438). The limiting effect of aromatic hydrocarbons on the process proposed by F. W. Richardson and J. L. Bowen (*J. Soc. Chem. Ind.*, 1908, **27**, 613) was indicated, and the intelligent application of Armstrong's method of polymerisation was advocated. It was shown that diluted sulphuric acid of the strength recommended by that author (*J. Soc. Chem. Ind.*, 1882, **1**, 480) had but little action on the turpentine substitutes then examined.

Richardson and J. E. F. Whitaker (*J. Soc. Chem. Ind.*, 1911, **30**, 115), in a later paper, agree with us that certain petroleums contain very appreciable amounts of benzene homologues, but apparently consider that only in Borneo petroleum is their amount worth taking into account. They state that the products of American petroleum are by far the most extensively used for adulterating turpentine, that for a year no Borneo spirit has been imported into Europe, and that they have never met with any turpentine substitute or turpentine adulterant composed of Borneo spirit.

In our experience, based on a knowledge of the materials in the London market, Roumanian spirit is by far the most extensively used for this purpose, although some American and Borneo spirit is also used. The reason is that the yield of "spirit" is greater (in the proportion of about 7 : 4) from Roumanian petroleum than from American, which, on the other hand, yields a better burning oil.

There is on the market a large quantity of pure Borneo spirit which is suitable for, and is used as, a turpentine substitute or adulterant. About 5,000 tons a year are imported, of which some 3,000 tons are used for this purpose. It is true, however, that no Borneo crude oil has been imported recently, as it is now distilled on the spot. The Borneo oil and spirit now being obtained are entirely different from that obtained a few years ago, the wells being sunk very much deeper and yielding a much lighter oil.

We agree that petroleum spirit, and not crude petroleum (by which is probably meant burning oil, crude petroleum being a thick brown liquid), is used to adulterate turpentine. The burning oil is not nearly sufficiently volatile for the purpose. American petroleum (burning oil) on distillation would only yield 90 per cent. at about 330° C.; whereas the petroleum spirit suitable for turpentine substitute would yield 90 per cent. at about 210° C., and an ordinary good turpentine substitute will yield 95 per cent. at about 210° C., and, in the case of Borneo spirit at a somewhat lower temperature.

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* Steuart (*J. Soc, Chem. Ind.*, 1900, **19**, 989). Poni (*ibid.*, 1906, **25**, 214 ; 1907, **26**, 958). Zaloziecki and Hausmann (*ibid.*, 1907, **26**, 1132). Edeleann (*ibid.*, 1908, **27**, 438). Jones and Wootton (*J. Chem. Soc.*, 1907, **91**, 1146).

Benzene, which is mentioned as a possible adulterant, should be out of the question, on account of its inflammability. Turpentine containing even 10 per cent. of benzene inflames at the ordinary temperature, and serious trouble with insurance companies and carriers would arise, to say nothing of the danger of making and using varnishes and paints with such spirit. For the same reason "gasolene," which Richardson and Whitaker also mention, and with which they have made various experiments, is an equally improbable adulterant. It is unlikely that spirit flashing below 73° F. is used for this purpose.

Richardson and Whitaker apparently realise that the use of a physical method of analysis is only possible in very restricted cases, when the nature of the constituents is accurately known and their amounts only are in doubt. They have supplemented the original method, which was probably sound in the simple case assumed by them by a chemical one which is certainly useful. They determine the "barium sulphate coefficient," which is a measure of the amount of sulphonation occurring when the liquid is treated with sulphuric acid under certain definite conditions. Their figures seem to show that under these conditions the sulphonation (of benzene) is not complete, as the amount of barium sulphate obtained is less even than that required for toluene. Experiments with Borneo and American petroleum and with American turpentine, which are quoted by them, show that whereas Borneo oil contains a large amount of constituents which can be sulphonated, American oil contains less, but still a notable amount, as does American turpentine (cymene, probably, formed by the action of the acid itself).

As no considerable amount of constituents boiling below 140° F. has been found by us, the presence of benzene or toluene is, apart from the commercial considerations already set forth, improbable in turpentine substitutes or adulterants, and, in our opinion, it is not desirable to return either of these hydrocarbons as present without direct proof of their presence. For these reasons we cannot consider the examples given by Richardson and Whitaker of analyses of mixtures containing benzene as in any way representing those likely to occur* in practice, nor do we think that with unknown constituents it would be possible to return analyses in the form given by them—*i.e.*, as percentages of turpentine, petroleum, gasolene, and benzene—but only as terpenes, homologues of benzene, and paraffins and cyclo-paraffins.

The separation from turpentine by fractional distillation of any adulterant likely to be employed is highly improbable of accomplishment, if not absolutely impossible.

In all cases where a sample of turpentine is suspected, its "close flash" (Abel test) should be taken. This figure for the pure article has not been found in the authors' experience to vary more than a few degrees, and all samples which they have examined have a close flash of 90° to 94° F. Any departure from these figures will make it necessary to examine the turpentine very closely. The simplest method, however, to determine whether or not the sample is pure is simply to shake it, when any froth will indicate that the turpentine is impure, and suggest the presence of petroleum adulterants (Nash, ANALYST, 1908, **33**, 231; 1909, **34**, 151). Coal-tar benzene does not give any froth, but, as previously stated, this could not be used.

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* We use advisedly the expression "likely to occur," since neither of us has for some years found an adulterated sample of turpentine.

Distillation of the sample will also give valuable information, as most turpentine substitutes and adulterants show a considerable rise in temperature towards the end of the distillation. Old and oxidised turpentine may also show a considerable rise, on account of the large amount of "residue" contained. A difference of as much as 13° C. has been noticed by one of us in the temperature at which the 95 per cent. fraction distilled in the case of two samples taken from bulk—deliveries of genuine American turpentine—the residue being respectively 4 per cent. and 1·9 per cent. An examination of the residue will show at once whether the rise was caused by petroleum spirit or by oxidation of the turpentine. We give the results of the examination of a few samples of turpentine substitutes obtained recently, which appear to be of special interest.

TABLE I.

	Borneo Spirit.		Roumanian Spirit.	
$D\frac{15\cdot5^\circ\text{ C.}}{15\cdot5^\circ\text{ C.}}$	0·8116	0·8190	0·8000	0·8060
N_D 20° C.	1·4548	1·4538	1·4432	1·4448
Flash-point (Abel)...	81° F.	95° F.	96° F.	97° F.
Behaviour on Nitration :				
Lower oily layer,* per cent. volume	60	52	30	28
Distillation (in Wurtz Flask) :				
10 per cent. over at	148·5° C.	159·0° C.	159·0° C.	165·5° C.
20 ,, ,, ...	150·0	160·5	161·0	167·0
30 ,, ,, ...	151·0	162·5	163·0	172·0
40 ,, ,, ...	152·5	164·5	165·5	175·0
50 ,, ,, ...	154·0	167·5	168·0	179·0
60 ,, ,, ...	156·0	170·0	171·0	183·0
70 ,, ,, ...	158·5	174·5	174·5	188·0
80 ,, ,, ...	162·0	180·5	180·0	193·0
90 ,, ,, ...	168·5	190·0	190·5	202·5
95 ,, ,, ...	174·5	200·0	203·0	212·0

These are typical of the materials now in common use as turpentine substitutes, and in physical and chemical properties are similar to other earlier samples examined by us (Coste, ANALYST, *loc. cit.*), and to several recently obtained from ordinary trade sources. They are obviously mixtures of aromatic and aliphatic hydrocarbons containing considerable proportions of the former class of bodies. It has been shown that such mixtures are very little acted on by diluted sulphuric acid of the strengths recommended by Armstrong; and as it appeared desirable to make certain that this behaviour was not affected by the presence of terpenes, the following series of experiments was made.

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* This is an approximate indication of the amount of aromatic hydrocarbons.

Measured quantities (from 100 to 200 c.c.) of mixtures of American turpentine and turpentine substitutes, such as are actually used, were treated with the diluted acids used by Armstrong for polymerisation, the treatment with the stronger acid being repeated as advised by him. The modification suggested by Morrell (*J. Soc. Chem. Ind.*, 1910, **29**, 241) was adopted, but in view of the probable presence of aromatic hydrocarbons treatment with strong acid was omitted, it being sought to confine the action to one of polymerisation. The results are given in Table II.

It was found in 1910 that the average loss suffered by turpentine substitutes when treated as above was 3 per cent. Armstrong states that about 5 per cent. of unpolymerisable liquid is left by pure American turpentine. If desired, the percentages of terpenes and of other hydrocarbons found can be corrected for these amounts. We give these corrected results in the third column. The correction is naturally less accurate when the proportion of either constituent is very small.

TABLE II.

	Volume Percentage of Adulterant.	Volume Percentage of Unpolymerised Distillate.	Corrected Volume Percentage of Adulterant.
I.	35	36·5	34·5
II.	12	15·2	11·5
III.	90	90·9	92·2
IV.	75	73·5	74·6
V.	16	19·3	15·8
VI.	5	8·2	3·7
VII.	20	23·0	19·8

These results confirm those previously obtained by us and those of Morrell (*loc. cit.*).

Finally we conclude that—

1. The substances most likely to be used to adulterate turpentine are those which are sold on a large scale as substitutes for it.

2. These are in most cases prepared from such varieties of petroleum as yield, in the fractions which are of suitable volatility, large proportions of aromatic hydrocarbons.

3. Such mixtures of hydrocarbons as are mentioned above are, either alone or in the presence of terpenes, but very slightly affected by sulphuric acid of a strength sufficient to effect the polymerisation of the normal constituents of American turpentine, and that this circumstance affords the best means of their separation* and the estimation of their amount.

4. The admixture of these substances with turpentine has such an effect on its volatility and surface tension as to render the flash-point† (Abel close test) and frothing test the best means of detecting their presence. The latter test also gives

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* We note that Richardson and Whitaker fully appreciate the *qualitative* value of this process.

† The close flash of turpentine substitutes has been found by us to range between 74° and 104° F., and therefore a normal "flash" does not necessarily preclude adulteration.

useful quantitative indications under certain circumstances (Nash, ANALYST, 1909, **34**, 151). Fractional distillation should also be carried out.

5. The proportion of aromatic hydrocarbons in mixtures from which terpenes have been removed can be estimated approximately by either a method of nitration, as used by Steuart, Jones, and Wootton and Coste; or one of sulphonation, as used by Richardson and Whitaker, and suggested also by Coste.

DISCUSSION.

Mr. CHAPMAN asked if the authors could say what was the general view held by turpentine users as to whether the presence of pine-wood oil—the oil distilled directly from the wood—should be regarded as an adulteration or not.

Mr. COSTE said that in the London County Council *v.* Holzapfel it was held that any mixture which contained a substance coming within the definition of petroleum laid down in the Petroleum Acts, and of which the flash-point was below 73° F., must be regarded as petroleum to which such Acts apply,* so that many of the adulterated samples referred to by Mr. Richardson could not lawfully be stored except under licence from the authority administering the Petroleum Acts, and therefore, as had been indicated in the paper, the conditions with which Mr. Richardson had to deal were somewhat unusual. With regard to the Armstrong process, their point was rather that the turpentine substitutes ordinarily sold at the present time contained large proportions of aromatic hydrocarbons, and that, this being the case, it was injudicious to employ any treatment with sulphuric acid that could cause the sulphonation of such hydrocarbons. Jacobsen and Ivan Levinstein had shown that ordinary concentrated sulphuric acid would dissolve, with sulphonation, both m- and o-xylene in the cold, and many other hydrocarbons were similarly sulphonated; so that, in using Armstrong's process for the purpose of determining modern turpentine substitutes in turpentine, one must be careful that the chemical processes going on were understood, or one might be sulphonating the hydrocarbons, and possibly forming additive compounds of any olefines that might be present.

Mr. NASH said that he had not found American turpentine coming into this country to be adulterated with pine-wood spirit. This spirit, when properly rectified, yielded an excellent white product, very similar in physical properties to American turpentine, and personally he should be inclined to use it almost as readily as American turpentine. Nevertheless, it was, of course, not turpentine, and if he found American turpentine to contain it he should feel bound to say that it was not pure American turpentine.

Mr. CHAPMAN asked if Mr. Nash had found the Herzfeld test for pine-wood oil to be satisfactory. He (Mr. Chapman) had recently met with some samples, which he believed to be pine-wood oil, which did not give the reaction.

Mr. NASH said that so far as he knew the Herzfeld test was satisfactory. A strong reaction was given by Finnish and Russian turpentine, or by any turpentine distilled from the wood. With regard to Roumanian spirit, he had been informed by some of the largest suppliers of turpentine substitutes in this country that the spirit they used was Roumanian spirit, the reason being that American petroleum yielded better burning oil than Roumanian, and also that Roumanian oil yielded far more spirit than American. He was surprised to hear from Mr. Richardson that benzene and gasoline were used in the North, because light spirits were much in demand for motors, etc., and the heavier kinds were much more suitable for turpentine substitutes.

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* This decision is adopted by Order in Council of May 7, 1907, whereby the Acts are made to "apply to any mixture of petroleum with any other substance . . ."—Statutory Rules and Orders, 1907, No. 483.

Messrs. F. W. RICHARDSON and J. E. F. WHITAKER send the following note: We are at a loss to understand the remark that no spirit flashing below 73° F.—hence no gasoline—is used, or that benzol, on account of its inflammability, is likely to be employed for the adulteration of turpentines; but Messrs. Coste and Nash say that they have not met with an adulterated sample of turpentine for some years, and as we have analysed a number of commercially adulterated turpentines we speak from actual experience.

We have analysed several so-called "turpentines" which flashed at the ordinary temperature and which contained gasoline. We only suggest the possibility of the use of benzol on account of its very low price—so low that motorists have turned to it as a cheap substitute for petrol. The adulterator does not concern himself with possible difficulties with insurance companies as long as he sells his material. We do not affirm the possibility of always being able to separate turpentine adulterants by mere fractional distillation. By using Young's still head we can absolutely separate gasoline and also benzol with fair accuracy from turpentine which has a much higher distilling temperature. In the absence of Borneo spirit and Roumanian spirit, hitherto not found by us in use as turpentine substitutes—an absence proved by the low barium sulphate coefficient—we do maintain that the Richardson and Bowen method of calculation, using the refractive indices of the fractional distillates, gives good results. American petroleum products are used in quite overwhelming proportion to any others for turpentine adulteration. In certain cases it would no doubt be advisable to classify the constituents of a mixture as described by Messrs. Coste and Nash; but most of the adulterated samples we have examined have allowed of a more popular grouping, "gasoline" and "petroleum" representing paraffins coming over on distillation at well-defined temperatures, the amounts being calculated from the data given by the distillates.

The term "cyclo-paraffins" might be very confusing to the non-chemical mind. The authors agree with us that "distillation of the sample will also give valuable information."

The Richardson-Whitaker methods allow the analyst to make a classification of the constituents of an adulterated turpentine in such a manner as to give a good general idea of the nature and amounts of the adulterants—not merely a division into turpentines and paraffins.

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"CONSTANTS OF CHICKEN AND TURKEY FATS."

By RAYMOND ROSS, F.I.C., and JOSEPH RACE, F.I.C.

(*Read at the Meeting, April* 5, 1911.)

In the following table a number of constants for chicken fat and turkey fat are given:

	Fat.		Fatty Acids.	
	Chicken.	Turkey.	Chicken.	Turkey.
Sp. gr., 100° F.	0·9065	0·9090	0·8866	0·8990
Koettstorfer	204·6	191·6	208·3	195·0
Molecular weight	—	—	270·0	287·0
Iodine value (Wijs)	71·5	66·4	73·6	70·7
Reichert Meissl	1·8	3·8	—	—
Polenske number	2·1	1·6	—	—
Acetyl value	—	—	25·4	18·4
(Ricinoleic acid, per cent.)	—	—	13·7	9·9
Hehner value	94·6	95·1	—	—
Melting-point, ° C.	23 to 27	31 to 38	27 to 30	37 to 38
Zeiss at 50° C.	47·5	46·0	36·5	32·5

Refractive Index, 50° C.:

Nd	1·45760	1·44854
Nc	1·45503	1·44592
Nf	1·46359	1·45433
Ng	1·46862	1·45952

The fats were optically inactive.

An examination of the fat from fowls which had died of overfeeding invariably showed a higher Zeiss number and iodine value than the normal values given above. This result is generally brought about by the use of too much heating food.

The formulæ used for calculating ricinoleic acid and triricinolein were respectively as under:

$$\text{Ricinoleic acid in fatty acids} = \frac{100\ \text{A}}{\text{A}\ (165 - \text{A})\ \frac{340}{298}}.$$

$$\text{Triricinolein in oil} = \frac{100\ \text{A}}{\text{A}\ (159{\cdot}1 - \text{A})\ \frac{1058}{932}}.$$

A = Acetyl value.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Composition of the Pulp of Cassia Fistula. L. C. Griebel. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 283-288.)—The results given in the following table were obtained on the analysis of various samples of cassia pulp:

	Water.	The Dry Substance contained—					
		Soluble Extract.	Insoluble Matters.	Invert Sugar.	Sucrose.	Sugar-Free Extract.	Ash.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Raw commercial pulp after removal of seeds and tissue ...	14·99	92·22	7·78	16·42	53·13	22·67	4·02
Raw pulp, free from seeds and tissue ...	19·89	90·01	9·99	6·79	61·13	22·09	5·67
Ditto	14·39	93·68	6·32	7·05	66·31	20·31	3·98
Purified pulp from same pods as No. 2	41·42	92·18	7·82	10·64	57·22	24·32	4·68
Pulp No. 4, plus 25 per cent. sucrose	33·27	93·81	6·19	7·12	72·36	14·33	2·91
Commercial purified pulp	25·99	92·42	7·58	51·81	26·25	14·36	2·82

These results show that the presence of added sucrose is most plainly indicated by the quantities of ash and sugar-free extract, the sucrose-content affording but little indication of the admixture. Sample No. 6 was of abnormal composition; it did not contain added sucrose or lactose, and the author is at present unable to give an explanation of the figures obtained. The aqueous extract of cassia pulp was found to contain, in addition to sucrose and invert sugar, citric acid, tannin, pectinous substances, and yellow and brown colouring matters. W. P. S.

Quantity of Husk and Germ in Cocoa Products. H. Huss. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 94-101.)—The author has examined a large number of samples of cocoa prepared in different factories, and finds that the better qualities of the product are practically free from husk and germ. He considers that manufacturers should have no difficulty in preparing cocoa-mass containing not more than 2 per cent. of husk and germ; the cocoa manufactured from such cocoa-mass would contain less than 3 per cent. of husk and germ. The manufacturers separate the husk and germ from the beans by means of machinery, and obtain products which consist of almost pure kernel, germ, and husk respectively. The table given below shows the amount of germ and husk present in the various fractions thus obtained, some manufacturers separating the cocoa into more fractions than do others:

	Factory 1. Trinidad Beans.		Factory 2. Bahia and St. Thomé Beans.		Factory 3. St. Thomé Beans.		Factory 4. Mixed Beans.		Factory 5. Trinidad and Caracas Beans.		Factory 6. St. Thomé Beans.	
	Husk per Cent.	Germ per Cent.	Husk per Cent.	Germ per Cent.	Husk per Cent.	Germ per Cent.	Husk per Cent.	Germ per Cent.	Husk per Cent.	Germ per Cent.	Husk per Cent.	Germ per Cent.
Fraction 1	1·3	0·05	—	—	1·6	0·01	0·38	0·005	0·12	0·25	1·60	0·05
Fraction 2	1·0	0·01	—	—	0·01	1·5	0·28	0·005	0·20	10·0	25·0	1·5
Fraction 3	1·8	0·20	—	—	—	—	0·40	2·0	—	—	—	—
Fraction 4	14·0	7·50	0·30	0·02	—	—	0·50	11·0	—	—	—	—
Fraction 5	35·0	—	0·75	5·65	—	—	—	—	—	—	—	—
Fraction 6	—	—	10·0	—	—	—	—	—	—	—	—	—

	Factory 7. Mixed Beans.		Factory 8. St. Thomé Beans.		Factory 9. Arriba Beans.		Factory 10. St. Thomé Beans.		Factory 11. —	
	Husk per Cent.	Germ per Cent.	Husk per Cent.	Germ per Cent.	Husk per Cent.	Germ per Cent.	Husk per Cent.	Germ per Cent.	Husk per Cent.	Germ per Cent.
Fraction 1	0·5	0·05	1·8	0·13	0·01	0·02	0·01	0·10	—	—
Fraction 2	20·0	30·0	3·0	5·0	1·50	8·0	7·57	0·45	2·0	15·0
Fraction 3	—	—	—	—	—	—	21·4	0·27	—	—
Fraction 4	—	—	—	—	—	—	8·0	2·0	—	—

The fractions containing much husk and germ are used only for the preparation of cheaper qualities of chocolate. The following method is described for the approximate estimation of husk and germ in cocoa; it depends on the fact that all the cocoa tissue, starch, etc., is dyed by the colour known as Brilliant Blue, whilst the mucilaginous cells of the husk and the tissue of the germ remain uncoloured. Congo red is also added, in order to dye particles of the inner skin of the bean which may be present, and the addition of the colour Soudan III. dyes the fatty droplets. The reagents employed are prepared as follows: Congo red, 1 per cent. aqueous solution; Brilliant Blue, 1 grm. of the colour dissolved in a mixture of 20 grms. of glycerol and 80 grms. of water; Soudan III., 0·1 grm. of the colour dissolved in a mixture of 50 grms. of glycerol and 50 grms. of 95 per cent. alcohol. A quantity of from 0·01 to 0·05 grm. of the cocoa or chocolate is rubbed down on a microscope slide with a drop of the Soudan solution, and the mixture is heated gently. A drop of the Congo red solution is now added, and, after the lapse of about one minute, 1 or 2 drops of the Brilliant Blue are well mixed with the mass. A hot cover-glass is then placed over the slide, and the specimen is examined under the microscope. It will be observed that the oil-drops are coloured reddish-yellow and the particles of the inner skin red. Starch cells are coloured blue. The husk and germ tissue which may be present are not coloured, and are readily detected in the blue or violet-coloured field. The quantity of husk and germ present may be estimated by comparing the slide with similarly treated specimens of cocoa containing known amounts of these two substances. W. P. S.

Estimation of the Doughing Value of Flour. A. A. Besson. (*Chem. Zeit.*, 1911, **35**, 245-246.)—Arragon has described two methods for the estimation of the "doughing value" of flour: first by the addition of increasing quantities of flour to a fixed quantity of water, secondly by adding water to a fixed quantity of flour. The second is the better method, and the first always gives higher values. The author has endeavoured to standardise the second method and to investigate the sources of error. If too much water be added all at once, the results are too high; if the water be added too gradually, they are too low. These errors are due to irregularity of composition of the dough. Very energetic kneading is necessary, using the whole hand, especially the palm, the dough being squeezed with the tips of the fingers pressing tightly against the palm. 150 or 200 grms. of flour are treated with 50 per cent. of water, added all at once, in a large porcelain basin. With the help of a glass stirrer a dough is made as stiff as possible. The rod is withdrawn, wiped with the hand, and the dough is kneaded to break down all dry portions, with the further addition of as much water as may be necessary, until all portions are uniformly wetted. The process is complete when a dough is obtained which no longer sticks to the fingers. If the first experiment shows that 50 per cent. of water is too much, the trial must be repeated with less. The dough is finally scraped together with a spatula and weighed. The results of three determinations should agree to within 1 per cent. of added water. Allowance should be made for variations in the percentage of moisture in the original flour, and results expressed in terms of dry substance. The laboratory estimations are accurately comparative, but in no way correspond with the actual proportions of water used in the practical preparation of baker's dough. For instance, a flour absorbing 45·6 per cent. of water in the laboratory was mixed with 72 per cent. in the bakehouse. J. F. B.

Detection of Fusel Oil in Brandy. E. Herzog. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 280-282.)—The reaction described by Hollander (*Chem. Zentralbl.*, 1910, **1**, 671) is found by the author to be simply a test for furfural and not for amyl alcohol; it is, moreover, not a very sensitive test for furfural. Benzaldehyde, fennel oil, and cinnamon oil also give a green coloration with the test.

W. P. S.

Cause of the Poisonous Properties of Certain Brands of German Margarine. W. Plucker. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 257-265.)—Many cases of vomiting and abdominal pains having been caused by the consumption of certain brands of margarine manufactured by a German firm, the author carried out a number of chemical and physiological experiments with the margarines, with the object of ascertaining the nature of the substance, present in the fats, which produced the above-mentioned symptoms. The active substance was found to be a non-volatile, hydroxy fatty acid; the physiological action of this fatty acid was similar to those of crotonic acid and an acid obtained from curcas oil. The margarine was optically active, a 10 per cent. solution showing a rotation of 0·70° in a 100 mm. tube; in this respect the margarine resembled croton oil but not curcas oil, which is inactive. The author has also examined another fat, cardamom or marotty fat, which acts as an emetic and is optically active. The objectionable properties of the margarine were most probably due to the presence of a quantity of one of the oils mentioned.

W. P. S.

Estimation of Potassium Nitrate in Meat. E. Polenske and O. Köpke. (*Arbeit. Kaiserl. Gesundheitsamte*, 1911, **36**, 291-296.)—The authors find that Stuber's modification of the Schlösing-Wagner method for the estimation of nitrates yields trustworthy results when applied to the estimation of saltpetre in meat; this method consists in converting the nitric acid into nitric oxide by boiling the nitrate solution (in this case the aqueous extract of the meat) with hydrochloric acid and ferrous chloride, and measuring the volume of the nitric oxide liberated. A similar method, proposed by Pfyl, in which the liberated nitric oxide is not measured, but oxidised with permanganate, the excess of the latter being then titrated, also gives accurate results, but Stuber's method is recommended as being more simple. The "nitron" method (ANALYST, 1905, **30**, 256), or the same as modified by Paal and Mehrtens (ANALYST, 1906, **31**, 405), and methods which depend on the conversion of the nitric acid into ammonia, yield inaccurate results, the former because of the interfering action of chlorides, which are not removed completely by lead acetate, and the latter because an aqueous extract of meat yields ammonia when distilled in the presence of an alkali or magnesia, even if nitrates be absent (*cf.* ANALYST, 1910, **35**, 206).

W. P. S.

Seychelles Cinnamon Bark. J. Meyer. (*Arbeit. Kaiserl. Gesundheitsamte*, 1911, **36**, 372-381.)—The following results were obtained in the analysis of a sample of cinnamon bark obtained from the Seychelles Islands:

	Ash.	Alcohol Extract.	Cinnamon Oil.	Crude Fibre.	The Cinnamon Oil contained—	
					Eugenol.	Cinnamic Aldehyde.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Mixed bark	5·50	10·37	1·12	38·1	—	—
Thin, young bark, 2·5 to 4 mm. thick	6·14	12·10	1·76	39·1		71·2
Thick, old bark, 7 to 12 mm. thick	5·00	9·50	0·90	37·5	14·5	68·2
Ditto, ditto: Inner layer	7·59	15·50	2·68	40·0	16·5	73·5
„ „ Middle layer	—	—	0·96	33·6	17·3	72·6
„ „ Outer layer	4·56	7·94	0·36	19·5	15·7	60·1

These results are very similar to those yielded by Ceylon cinnamon. The odour of the oil is somewhat different to that of the oil obtained from Ceylon cinnamon, the latter oil containing from 4 to 8 per cent. of eugenol, and from 65 to 75 per cent. of aldehyde. The chief difference between these two kinds of cinnamon lies in the fact that Ceylon cinnamon is practically freed from the outer bark before exportation. This cork layer found on the Seychelles bark is almost worthless as a spice, but the other portions of the bark are valuable. W. P. S.

Detection of the Use of Alkali Carbonates in Cheese Manufacture. H. Höft. (*Milchwirtsch. Zentralbl.*, 1911, **7**, 25-31.)

Composition of Goats' Milk, and the Detection of Goats' Milk in Cows' Milk. H. Hager. (*Milchwirtsch. Zentralbl.*, 1911, **7**, 19-24.)—The following were the average results obtained on the analysis of 200 samples of goats' milk: Sp. gr., 1·0324; fat, 3·8 per cent.; solids-not-fat, 9·02 per cent. The presence of not less than 10 per cent. of goats' milk in cows' milk may be detected by the behaviour of the casein towards concentrated ammonia; the latter dissolves the casein of cows' milk, whilst the casein of goats' milk simply swells up without dissolving.

Preservation of Fruit Juices with Hydrofluoric Acid. R. Cohn. (*Zeitsch. öffentl. Chem.*, 1911, **17**, 2-12.)

Adulteration of Linseed Cakes and Linseed Meals in Belgium. L. Bussard. (*Ann. Falsif.*, 1911, **4**, 30-31.)—The principal adulterants found were rice meal, cottonseed meal, earthnut meal, linseed offal, and sand.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Relative Proportions of Arsenic in Marine Algæ and Products derived from them. E. Tassilly and J. Leroide. (*Bull. Soc. Chim.*, 1911 [iv.], **9**, 63-66.)—The authors confirm the conclusion of Gautier (*Bull. Soc. Chim.*, 1903 [iii.], **29**, 33 and 863), that arsenic is a normal constituent of certain algæ, though a small proportion of the arsenic found in commercial products derived from them no doubt has its origin in the chemicals used in their manufacture. These commercial processes are partly secret; but when they are imitated so far as is possible, using chemicals which are either free from arsenic or of which the arsenic content is very small, and known accurately, it is found that the whole of the arsenic in the algæ finds its way into the finished product. The following figures are given to show the proportion of arsenic in certain species of marine algæ and fungi, the proportion for any one species being fairly constant. The figures represent mgrms. of arsenic per 100 grms. of the samples, which contained 20 to 30 per cent. of water, and had been washed with water until the washings were free from chloride:

Chondrus crispus	0·070
Fucus vesiculosus	0·010
Corsican moss	0·025
Laminaria digitata	0·050
,, *saccharina*	0·010
,, *flexicaulis*	0·010

G. C. J.

Bacteriological Investigation of Soil. T. Remy and G. Rösing. (*Zentralbl. f. Bakter. u. Parasitenk.*, 1911, 29, 36-77; through *Chem. Zentralbl.*, 1911, 1, 921-922.)—It was found that the composition of a soil had an unmistakable influence upon the decomposition of peptone, both vegetable nutrient substances and other constituents of the soil playing a part in the process. Thus the mixture of humic compounds and silica, obtained by extracting the soil with hydrochloric acid, has a very stimulative effect upon the decomposition, although in many soils there appear to be compounds soluble in water which have a restrictive influence. This influence of the soil constituents is not so pronounced, however, as entirely to outweigh the influence of the biological factors. Under the conditions of the experiments loam had a much greater decomposing action than river sand upon peptone. The intensity of the decomposition of peptone and the putrefaction of albumin, both in soil and in nutrient solutions, stood in manifest relationship to the degree of aeration. Thus, in drawing any conclusions as to the biological capacity of the soil from the degree of decomposition of peptone, the values obtained only hold good for the particular conditions of the experiment. For testing the putrefactive capacity of a soil, nutrient media are prepared containing 1 per cent. of pure gelatin, 0·1 per cent. each of potassium phosphate, magnesium sulphate, and sodium carbonate, and the sterilised aqueous extract of 10 per cent. of the soil. The nutrient solution thus obtained is divided into portions of 50 c.c., and inoculated with the extract obtained by shaking 0·1 to 0·25 grm. of the soil in physiological salt solution. The cultivation temperature should not exceed 20° C. After seven or eight days an estimation is made of the amount of nitrogen displacable by magnesium oxide, but there is no perceptible loss of total nitrogen during the decomposition. More decisive data may be obtained by determining the course of putrefaction in a small sample of the soil. For this purpose the slightly dried soil is mixed with 1 per cent. of gelatin, and introduced in quantities of 500 to 1,000 grms. into covered glass basins, the proportion of moisture being then maintained at about 60 per cent. of the water capacity as estimated by Wahnschaffe's method. The basins are allowed to stand for a week at about 20° C., and the total and ammoniacal nitrogen in the soil then estimated. It might also be advantageous to estimate the amount of nitrogen soluble in water saturated with carbon dioxide in the manner devised by Mitscherlich.
C. A. M.

Bacteriological Method of Estimating Available Organic Nitrogen. J. M. McCandless and F. C. Atkinson. (*J. Ind. and Eng. Chem.*, 1911, 3, 174-175.)—The method employed depends upon the observation of the amount of ammonia produced by the action of soil bacteria on the soluble and insoluble proteins of the material under examination (*cf.* also Lipman, *J. Ind. and Eng. Chem.*, 2, 146). One grm. of the sample is washed with hot water on a filter, and the filtrate made up to 500 c.c. The total nitrogen, and nitrogen as free and saline ammonia, are estimated on portions of this solution. A known quantity of soil is next shaken up with the aqueous extract of the material, and a series of definite portions of the filtered liquid incubated for 210 hours at 38° to 40° C., both under aerobic and anaerobic conditions. In the latter case the liquids remain practically free from nitrates and nitrites. The insoluble residue from the sample is also incubated with 50 c.c. of a liquid containing soil bacteria, prepared by shaking the soil with a solution containing 0·1 per cent. potassium phosphate, 0·2 sodium chloride, 0·05 magnesium sulphate, and 0·005 calcium chloride. This incubation is extended over twenty-six days, the daily increase in soluble ammonia being determined by using one of the series of flasks prepared. The authors' experiments show that the bacteria concerned convert the insoluble proteins first into a soluble form, subsequently decomposing the soluble substances into ammonia. Experiments with cottonseed meal, for example, containing 8·20 per cent. of total nitrogen expressed as ammonia, showed this material to contain nitrogen (as NH_3) 1·01 per cent. in the aqueous solution, of which 0·73 per cent. was water-soluble organic nitrogen (as NH_3). Incubation for 210 hours as described produced 0·22 per cent. of ammonia from the water-soluble organic nitrogen, and 2·09 per cent. nitrogen (as NH_3) remained insoluble after twenty-six days' incubation, while 5·10 per cent. was rendered soluble by this treatment. A. R. T.

Biological Examination of Honey (Enzymes). E. Moreau. (*Ann. Falsif.*, 1911, 4, 65-66.)—The presence of catalase, amylase, and invertase, was detected in twenty samples of French honey examined, but anaeroxydases and aeroxydases were absent. The largest quantities of catalase were present in those honeys which contained considerable amounts of proteins, from 4 to 6 c.c. of oxygen being liberated from hydrogen peroxide in twenty-four hours by the action of the catalase present in 5 grms. of the honey. With other samples the volume of oxygen liberated varied from 0·3 to 0·7 c.c. The honeys containing most catalase also contained the greater quantities of amylase, as was shown by their action on starch paste. The invertase was detected by means of its action on sucrose, the enzyme being first precipitated from the honey with alcohol. (*Cf.* ANALYST, 1910, **35**, 165, 259.) W. P. S.

Method for the Estimation of Small Variations in the Quantity of Calcium contained in Blood. N. Voorhoeve. (*Biochem. Zeitsch.*, 1911, **31**, 195-206.)—The method proposed depends on the fact that blood does not coagulate when all the calcium present has been precipitated by the addition of ammonium oxalate. In carrying out the process, a small syringe is one-half filled with a 0·9 per cent. sodium chloride solution containing 229 mgrms. of crystallised ammonium oxalate per 100 c.c.; the needle of the syringe is then inserted into a vein and the other half of the syringe is filled with blood, the total volume of the syringe being 5 c.c. The mixture of blood and sodium chloride-ammonium oxalate solution is now transferred from the syringe to a watch-glass, well mixed, and kept for twenty-four hours in an atmosphere saturated with water vapour. Portions of $\frac{1}{2}$ c.c. are then removed to a number of watch-glasses and mixed with different quantities of a calcium chloride solution containing 30 mgrms. of calcium oxide per 100 c.c. The watch-glasses are then placed in a chamber the atmosphere in which is saturated with water vapour, and after the lapse of twenty hours the contents of the glasses are examined. The point at which coagulation just takes place having been found, the quantity of calcium in the blood is easily calculated, since 100 c.c. of the ammonium oxalate solution are capable of precipitating 90 mgrms. of calcium oxide, and the quantities of calcium oxide introduced as calcium chloride solution are known. The coagulating point may also be observed by drawing the mixtures of oxalate-blood and calcium chloride into capillary tubes; these are placed horizontally for twenty hours, after the ends have been sealed with paraffin wax, and then examined in a vertical position. If coagulation has not taken place, the red corpuscles settle to the lower end of the tubes. W. P. S.

Note on the Bulgarian Bacillus. J. Effront. (*Comptes rend.*, 1911, **152**, 463-465.)—Bacilli isolated by the author from six medicinal preparations of lacto-bacilline differ in their chemical action on milk and peptone from the Bulgarian bacillus of the Pasteur Institute. The latter acidifies milk strongly, forms little volatile acid, and has but a slight action on casein even after thirty days' incubation; whereas lacto-bacilline organisms are strongly proteolytic and produce far less acid, most of which is volatile, and leave the milk as a clear yellow solution. The Bulgarian bacillus was not present in any of the medicinal preparations examined, all of which showed strong proteolytic activity. The author considers the organisms to be really alike, the above differences being due to biochemical variation arising from the conditions of culture. The Bulgarian bacillus retains all its characters when grown in normal milk, but is much changed by variation in the aeration and acidity of the medium. Thus, in a culture containing peptone, calcium carbonate, and glucose, the Bulgarian bacillus behaves like a normal lactic ferment till all the sugar is used up. If at this stage the whole is kept for a fortnight, a spore film-formation occurs, and the culture then gives all the reactions of the lacto-bacilline preparations. The author points out that the therapeutic applications of the Bulgarian bacillus have been made with pharmaceutical preparations, such as the lacto-bacillines examined, and not with the true Bulgarian bacillus. The effects obtained cannot, therefore, be attributed to the lactic acid, which is hardly produced at all, but are due to the proteolytic action. H. F. E. H.

Behaviour of Schardinger's Reagent towards Colostrum. R. Reinhardt and E. Siebold. (*Biochem. Zeitsch.*, 1911, **31**, 294-320.)—The milk (colostrum) yielded by cows immediately after calving, and frequently that yielded during the next few days, decolorises Schardinger's reagent (formaldehyde and methylene-blue; *cf.* ANALYST, 1903, **28**, 32), although somewhat feebly. The first true milk from the cow, however, does not as a rule decolorise the reagent, and only after the lapse of from three to eight weeks after calving does the milk again give a positive reaction. This period of inactivity exhibited by the milk varies greatly in length with different cows; the reason is not known, but it is ascertained that the suckling of the calf does not cause the variation. The amount of enzyme, reductase, in a milk is not dependent on the quantity of fat present, and it is never quite absent from milk at any part of the lactation period. Milk mixed with colostrum decolorises the reagent more rapidly than does the same milk when mixed with water, and the difference in behaviour of milk at the beginning and end of lactation towards the reagent is merely due to the amount of enzyme present, and not to the varying activity of the enzyme. A definite quantity of the enzyme is necessary to decolorise a given amount of the reagent. Illness of the cow and inflammation of the udder influence the reaction. The first milk drawn from a cow contains less of the enzyme than does that obtained at the end of the milking operation, and different teats of the same cow may yield quantities of milk containing different amounts of the enzyme; as a general rule, however, 10 c.c. of milk from a healthy cow at the latter part of the lactation period will decolorise 1 c.c. of the reagent within from four to twelve minutes.

The enzyme is formed in the udder, and milk obtained under sterile conditions from a cow behaves exactly the same towards the reagent as does that obtained by ordinary milking. Temperatures above 65° C. destroy the activity of the enzyme. The highest temperature at which the enzyme decolorises the reagent is 65° C. in the case of milk yielded towards the end of lactation, and 45° C. for milk at the beginning of the lactation period (ANALYST, 1909, **34**, 352). The presence of bacteria in milk causes, after a time, an increase in the activity of the milk towards the reagent, this being due to the production of enzymes by the bacteria. W. P. S.

On the Peroxydase of Cow's Milk and its Detection by Means of the p.phenylenediamine Test. E. Nicolas. (*Bull. Soc. Chim.*, 1911 [iv.], **9**, 266-269.)—Bordas and Touplain have expressed the opinion that the coloration obtained when milk is treated with certain reagents (guaiacum, p.phenylenediamine, etc.), in the presence of hydrogen peroxide, may be attributed to the action of the casein on the hydrogen peroxide, and not to the presence of a peroxydase in the milk. The author, however, shows that, whilst precipitated and boiled casein is capable of producing a blue coloration with p.phenylenediamine and hydrogen peroxide, it is the hydrogen peroxide which first reacts with the p.phenylenediamine, and that the casein only combines with the product formed to give the blue coloration. This reaction only takes place between the hydrogen peroxide and the reagent when the former is used in the form of a concentrated solution. The coloration is not obtained with casein if only 2 drops of hydrogen peroxide diluted previously with nine times its volume of water be used, but the addition of a trace of a solution containing a peroxydase (aqueous extract of bran) causes the immediate development of the coloration. It is therefore important, in applying the test for the purpose of distinguishing between heated and unheated milk, to employ diluted hydrogen peroxide.

W. P. S.

Hæmoglobin Preparations. M. Vogtherr. (*Zeitsch. öffentl. Chem.*, 1911, **17**, 41-45; through *Chem. Zentralbl.*, 1911, **1**, 1013.)—In the case of hæmoglobin preparations two conditions are of special importance: the hæmoglobin must be readily and completely soluble in water, and must preserve its physiological and chemical qualities as far as possible unaltered; for instance, it must not have undergone decomposition into hæmatoglobulin and hæmatin. Further, the hæmoglobin should be present, as far as possible, in the form of oxyhæmoglobin; this should not have suffered undue conversion into methæmoglobin and hæmochromogen. Analytical determinations comprise estimations of water, ash, iron, nitrogen, solubility and the observation of the spectroscopic characters. Among a series of samples examined, one preparation showed the best values for nitrogen and the proportion of blood-colouring matter; the unground modification showed the smallest alteration as regards the oxygen constituent. Hæmoglobin carbonate preparations are distinguished by excellent solubility, but undergo decomposition under the action of carbon dioxide. The percentage of moisture in the samples ranged from 5·94 to 7·92. In 100 parts of dry substance were found: Nitrogenous matter, 90·54 to 97·07; non-nitrogenous matter, 0·06 to 6·12; ash, 2·87 to 4·37; pure iron, 0·283 to 0·486. Many commercial hæmoglobin preparations have a basis of cacao, as powder or cake; others are as solutions of hæmoglobin, containing from 47 to 76 per cent. of water. J. F. B.

Chemistry of Honey Formation. M. Kustenmacher. (*Biochem. Zeitsch.*, 1911, **31**, 237-254.)—The physiology of the bee is described by the author, who shows that the nectar of flowers, which is an aqueous solution of sucrose containing other carbohydrates, tannin, organic acids, etc., is converted by the bees into honey. This change is brought about by the aid of invertase, which is obtained from the pollen of the flowers; the saliva of the bees does not introduce this enzyme into the honey, nor does the saliva increase its action. The principal difference between different kinds of honey is due to the aromatic constituents of the plants from which the nectar has been obtained; the quantity of tannin has also some influence on the character of the honey. W. P. S.

Relation of the Normal Yellow Pigmentary Substances of Urine to the Diazo-Reaction, and a Colorimetric Method for Estimating Urochrome and Urochromogen. M. Weisz. (*Biochem. Zeitsch.*, 1911, **31**, 333-356.)—The portion of urine which is precipitated by barium hydroxide and alcohol—that is, the proteic acid fraction—when treated with basic lead acetate, yields a precipitate containing the normal yellow pigment (the urochrome of Thudichum and Garrod) and the urochrome which Dombrowski obtained directly by precipitation with copper acetate. The two pigments are not identical, and may be separated from one another by taking advantage of the insolubility of Dombrowski's urochrome in dilute acetic acid. The nature of the substance which produces Ehrlich's diazo-reaction is not known; it is contained in the proteic acid fraction, and from its properties is termed a urochromogen. Another urochromogen is also present in urine, but this does not give the diazo-reaction; both urochromogens may be converted by oxidation into urochrome. The urochrome may be estimated in urine by treating 25 c.c. of the urine with 20 grms. of powdered ammonium sulphate, filtering off the other colouring matters which are precipitated, and comparing the filtrate in a Dubosq colorimeter with a standard solution of a yellow dye. The quantity of urochrome is expressed in terms of the standard colour. Urochromogens may be estimated in a similar manner after oxidation to urochrome. The occurrence of urochromogens in urine indicates toxic changes in the metabolism of the body. W. P. S.

Separation of Urobilin from Urobilinogen. L. Grimbert. (*Comptes rend.*, 1911, **152**, 727-728.)—The simplest method of detecting the chromogen of urobilin (urobilinogen) in urine is to extract it with chloroform, and to oxidise it to urobilin, which will then give a green fluorescence on adding an alcoholic solution of zinc acetate to the chloroform solution. (*Cf.* ANALYST, 1910, **35**, 367.) Urobilinogen may also be detected by gently heating 1 c.c. of the chloroform solution with 3 or 4 drops of Ehrlich's reagent (2 per cent. alcoholic solution of p.dimethylaminobenzaldehyde mixed with an equal volume of hydrochloric acid), and then adding 0·5 c.c. of 95 per cent. alcohol to obtain a homogeneous solution. In the presence of urobilinogen the liquid becomes reddish purple, whereas urobilin gives no coloration under the same conditions. Although this reaction is also given by indol, it is useful for studying the behaviour of the chromogen in the urine. It is only exceptionally that free urobilin derived from the oxidation of the urobilinogen is found in urine; and, as a rule, when this oxidation has taken place, the urobilin enters into combination with other substances, probably of an alkaline nature, to form a compound insoluble in chloroform, so that, in general, a simple extraction of the urine with chloroform will effect a separation of the two compounds. If, however, as is frequently the case, the urine has been acidified before applying the tests, the liberated urobilin will also be extracted by the chloroform. It may then be removed by shaking the chloroform with a neutral aqueous solution of disodium phosphate, the urobilinogen being then left in the chloroform. In testing urine the liquid is extracted with chloroform, and the extract filtered through cotton-wool, and treated drop by drop with an alcoholic solution of zinc acetate (1 : 1,000) until the initial turbidity has disappeared. The production of a green fluorescence indicates the presence of urobilin. In this case the chloroform is shaken with a few c.c. of disodium phosphate solution (neutral to phenolphthaleïn), and the chloroform layer (which no longer fluoresces with zinc acetate) is mixed with a trace of iodine. Urobilinogen, if present, will thus be oxidised to urobilin, and the chloroform will again show the green fluorescence with zinc acetate. C. A. M.

ORGANIC ANALYSIS.

Direct Estimation of Caoutchouc as Tetrabromide. W. Hinrichsen and E. Kindscher. (*Chem. Zeit.*, 1911, **35**, 329-330.)—The authors have investigated the properties of purified caoutchouc tetrabromide. Kassai caoutchouc was purified by repeated solution in benzene, precipitation of the filtered solution by alcohol, and extraction of the precipitate by acetone. The purified material was then brominated by Hübener's method, repeated treatment with bromine water being employed until the product could be readily reduced to powder in a mortar. After washing with water, the brominated caoutchouc was exhaustively washed with alcohol and then with ether. Caoutchouc tetrabromide, particularly that from vulcanised material, is readily soluble in chloroform, but the tetrabromide from raw caoutchouc leaves a slimy residue which is not completely soluble. From the chloroform solution the pure tetrabromide may be precipitated quantitatively by petroleum spirit. On storage with exposure to light and air, the dry purified tetrabromide gradually changes and becomes less soluble in chloroform.

The authors have found that when caoutchouc tetrabromide is decomposed by nitric acid in presence of excess of silver nitrate for the volumetric estimation of the bromine, the results obtained are much too low. This is due to the volatilisation of bromine compounds, and the loss of bromine is greater the higher the concentration of the nitric acid. The volumetric process recommended by Büdde and by Hübener is therefore unsuitable. As an alternative, the method described by Marcusson and Doescher (Analyst, 1910, **35**, 319), depending on the direct combustion of the substance in a current of oxygen and absorption of the products in standard silver nitrate, gives excellent results. As this requires special apparatus, the tetrabromide may be burnt in a platinum crucible with a mixture of potassium and sodium carbonates, care being taken to boil the precipitate of silver bromide, subsequently obtained, thoroughly with nitric acid. The authors provisionally indicate the following scheme for the analysis of caoutchouc by a modification of the tetrabromide method: Bromination should only be effected after the material has first been thoroughly extracted with acetone. The bromide is collected on a filter, together with the inorganic compounds remaining in it, washed with water, alcohol, and ether, and then dissolved in chloroform. The tetrabromide is precipitated from the chloroform solution by petroleum spirit, and is either dried and weighed directly, or the bromine is estimated by either of the methods described above. The direct weighing is preferable, as the substance is then available for the estimation of the sulphur of vulcanisation.

J. F. B.

Examination of Asphaltum. J. Marcusson. (*Chem. Rev. Fett- u. Harz-Ind.*, 1911, **18**, 47-51.)—The following modification of the author and Eickmann's test for distinguishing between natural and artificial asphaltum (Analyst, 1908, **33**, 476) enables very small quantities of petroleum pitch to be detected: 10 grms. of the sample are dissolved in 15 c.c. of benzene, the solution poured into 200 c.c. of petroleum spirit (b. p. 80° C.), the precipitated asphaltene separated, and the filtrate treated with three successive portions of 15 c.c. of concentrated sulphuric acid to remove the remaining dark-coloured constituents. The liquid is washed with alkali solution and with water until free from acid, and then evaporated, and the residue dried and weighed. In the case of natural asphaltum the residual oil (from 2 to 34 per cent. upon the ash-free material) will be fluid at 20° C., and show no separation of particles of paraffin wax or substances resembling vaseline; whilst in the presence of petroleum residues it will have the consistency of cylinder oils at 20° C., will contain an abundance of vaseline substances, and will amount to 26 to 58 per cent. of the ash-free sample. In doubtful cases an estimation of the paraffin wax in the oily residue will afford valuable information, the amount being less than 1 per cent. in the case of natural asphaltum, and ranging from 3·3 to 16·6 per cent. in the case of petroleum pitch. Thus, if the residue yielded more than 2 per cent. of paraffin wax, the presence of petroleum pitch is indicated. A further test may be based upon the fact that natural asphaltum yields considerable quantities of organic acids on distillation, whereas in the case of petroleum pitch the distillates contain only a slight amount of acids. In applying the test 30 grms. of the sample (or a quantity of material containing about 30 grms. of bitumen) are distilled in a small retort, and two fractions of 4 to 5 c.c. collected. These are dissolved in ether, the solution washed with water to remove any mineral acid, and the acid value estimated in the usual manner. In the case of natural asphaltum the first distillate will show an acid value considerably over 1, whilst that from the petroleum residues will show a value below 1. The second distillate from the natural asphaltum will also still have a considerable acid value, whereas in the case of petroleum residues it is invariably free from acid. Thus the following results were obtained:

	Natural Asphaltum.			Petroleum Residues.			
	Trinidad.	Syrian.	San Valentino (Soft).	From Heavy Wietzer Crude Oil.	Soft Pitch.	Galician I.	Galician II.
Distillate I....	14·0	4·7	1·9	0·53	0·73	0·57	0·87
,, II....	5·6	2·7	1·4	nil	nil	nil	nil

If a sample contained less than 1·7 per cent. of sulphur (upper limit for petroleum residues and lower limit for natural asphaltum), and also considerable quantities of paraffin wax, the presence of petroleum pitch is proved. If, further, the first distillate had an acid value exceeding 1, whilst the second distillate also showed a high acid value, and there was also a considerable amount of sulphur, the sample probably contained natural asphaltum in addition to petroleum pitch. The correctness of these deductions, however, depends upon the absence of fat-pitches (the presence of which is indicated by the production of acrolein on heating), which also give high acid values in the first distillate. In addition to these kinds of asphaltum there are also upon the market varieties obtained by distilling off considerable quantities of oil from naturally occurring soft bitumens. The characteristics of two of these thickened products were as follows:

Commercial Name.	Ash.	Sulphur.	Oily Constituents.			Acid Value of—	
			Nature.	Amount.	Paraffin Wax.	Distillate I.	Distillate II.
	Per Cent.	Per Cent.		Per Cent.	Per Cent.		
Mexico asphaltum	0·2	5·8	Not fluid. Contained vaseline-like particles	35·0	2·8	1·6	0·9
Derna asphaltum-bitumen	5·6	0·9	Ditto.	25·0	1·6	0·7	0·7

These products may thus be readily distinguished from petroleum residues. If present in admixture with petroleum pitch, Mexico asphaltum would be detected by its high proportion of sulphur and the acid values of the distillates. Derna asphaltum, however, would only be detected by these values when present in larger proportion than the petroleum residues in the mixture. The phenols in brown coal-tar pitch, to which Graefe's diazobenzene chloride reaction (ANALYST, 1906, **31**, 204; 1908, **32**, 477) is due, may be extracted by treatment with sodium hydroxide solution. Ordinary coal-tar pitch behaves in a similar manner, but may be distinguished from brown coal-tar pitch by other reactions (presence of free carbon, anthraquinone test, etc.). Wood tar also gives Graefe's reaction, but is easily distinguished from brown coal-tar pitch by its pronounced solubility in alcohol and its ready absorption of sulphur. When a positive result has been obtained in Graefe's reaction, the following test may be applied to determine whether the sample consisted of brown coal-tar pitch or a natural asphaltum, thinned by the addition of brown coal-tar oil (paraffin oil): Ten grms. of the asphaltum are dissolved in 15 c.c. of warm benzene, the solution poured into 200 c.c. of petroleum spirit, and the precipitate collected, washed with petroleum spirit, dried, powdered, and tested for phenols. In order to extract the latter as completely as possible, the powder is boiled for fifteen minutes beneath a reflux condenser with ½ N-alcoholic potassium hydroxide solution, after which the liquid is cooled and filtered, the filtrate evaporated to expel alcohol, and the residue taken up with water. The solution is shaken with sodium chloride to remove colouring matters, and the filtrate tested in the usual way with diazobenzene chloride solution. An addition of 10 per cent. of brown coal-tar pitch, or of 20 per cent. of acid resins, to natural asphaltum could thus be detected with certainty. Mixtures of natural asphaltum with 10 and 20 per cent. of brown coal-tar oil gave a sharp diazo reaction, but when the test was applied to the constituents insoluble in petroleum spirit no colour reaction was obtained. C. A. M.

Use of Carbalkyloxy Derivatives for the Quantitative Estimation of Hydroxyl Groups. K. C. R. Daniel and M. Nierstein. (*Ber.*, 1911, **44**, 701-704.)—Following E. Fischer and Freudenberg's observations on the properties of carbalkyloxy compounds of tannins, the authors have based a method for the estimation of hydroxyl groups in substances of the tannin class on the saponification of these derivatives, the preparation of which offers the advantage that intramolecular changes and splitting up of the sensitive tannin compounds are apparently excluded. The saponification takes place according to the equation—

$$R.O.COOR_1 + H_2O = R.OH + CO_2 + R_1OH,$$

and the quantitative estimation consists in weighing the carbon dioxide split off. The apparatus consists of a distillation flask with dropping funnel, supported in an oil bath and connected with an upwardly inclined Liebig's condenser, the further end of which is connected with a calcium chloride U-tube which leads to the absorption bulbs. Saponification is effected by means of boiling 50 per cent. pyridine, and the U-tube contains a mixture of 2 parts of calcium chloride and 1 part of oxalic acid to absorb any pyridine vapours. For the estimation, 0·3 to 0·5 grm. of the substance, dissolved in 20 to 30 c.c. of alcohol, is placed in the flask, which is heated in the oil bath at a temperature of 115° to 120° C., whilst a current of air free from carbon dioxide is passed through the system. The dropping funnel is slightly warmed, and 80 c.c. of a 50 per cent. solution of pyridine are introduced through it in three portions.

Heating is continued at a maximum temperature of 120° C. with continuous passage of air for three-quarters to one hour. The results of the analysis of a number of carbalkyloxy derivatives of various hydroxylated compounds illustrate the accuracy and convenience of the method. J. F. B.

Influence of Inactive Substances on the Rotation of Lævulose. N. Wender. (*Biochem. Zeitsch.*, 1911, **31**, 357-373.)—Most inorganic acids, when added to solutions of lævulose, increase the specific rotation of the sugar, the increase being in proportion to the acidity of the solution, whilst organic acids vary in their influence. Oxalic acid increases the rotation and acetic acid diminishes it. Alkalis depress the rotation owing to decomposition of the lævulose. Inorganic salts may increase or decrease the rotation, and alcohols and acetone always lower it, the diminution being proportional to the quantity of alcohol or acetone present.
W. P. S.

Characters of Creosote and Tar Oils available for Wood Preservation. C. N. Forrest. (*J. Soc. Chem. Ind.*, 1911, **30**, 193-196.)—A mixture of water-gas tar, freed to some extent from light oils, with coal- and coke-tar distillates, forms the bulk of American creosote of the present time. The simple oils and tars which are now available have the following characters:

	Domestic Distilled Oil.	Foreign Distilled Oil.	Otto Coke-Oven Tar.	Otto Coke-Oven Distilled Oil.	Water-Gas Tar Refined
Sp. gr. 15° C.	1·03	1·04	1·16	1·09	1·13
Free carbon	none	none	5·30	none	0·60
Tarry matter	0·5 to 2·0	0·70	58·00	10·0	51·00
Tar acids	7·0	13·60	trace	trace	none
Solid naphthalene ...	—	18·00	—	—	—
Distillation :	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent
Below 170° C.	0·42	0·70	0·60	1·60	0·50
170 to 205° C.	4·50	1·50	0·90	2·40	0·50
205 to 235° C.	51·95	35·20	6·90	19·80	1·70
235 to 255° C.	22·01	32·60	7·20	7·70	4·10
255 to 270° C.			4·20	7·50	5·80
270 to 315° C.	10·99	20·00	7·90	11·60	15·00
Residue	10·13	10·00	72·30	49·40	72·40
	100·00	100·00	100·00	100·00	100·00
Insoluble in dimethyl sulphate	none	none	none	none	none
Condition of residue ...	soft	soft	hard	soft	hard
Paraffin scale	none	none	none	none	none

The following table shows the analytical characters of three classes of materials extensively used for the treatment of wood-paving blocks :

	Water-Gas Tar.	Coal-Tar.	Distilled Oil.
Sp. gr. 38° C.	1·12	1·17	1·10
Insoluble in CS_2	0·60	3·5	none
Distillation :	Per Cent.	Per Cent.	Per Cent.
Below 170° C.	0·50	0·10	—
170 to 205° C.	0·50	2·00	—
205 to 235° C.	1·70	2·90	—
235 to 255° C.	4·10	7·30	—
255 to 270° C.	5·80	7·20	—
270 to 315° C.	15·00	9·70	25·00
315 to 370° C.	24·60	16·40	60·00
Residue	47·80	54·40	15·00
	100·00	100·00	100·00
Insoluble in dimethyl sulphate ...	none	none	none
Condition of residue	hard	hard	soft
Sp. gr. distillate, (255 to 315°) at 60° C.	1·00	1·02	1·08

The following characters are relied on for the determination of the suitability of preservative material for a specific purpose : Sp. gr., preferably at 38° C. ; free carbon = insoluble in benzene or carbon disulphide ; pitch or tarry matter = insoluble in alcohol or petroleum naphtha ; tar acids = volume removed from distillate by caustic soda solution ; naphthalene = solid from distillate or fraction 210 to 235° C. ; distillation (under standard conditions) ; sp. gr. of certain or all fractions of distillate ; sulphonation and dimethyl sulphate tests of distillate ; refractive index of distillate. Cold solvents for pitch and carbon determinations are advantageous and generally accurate. Tar-acids are not of importance. Solid naphthalene should be pressed from the chilled distillates. As regards distilling apparatus, the method recommended by Von Schrenk, Fulks and Kammerer (*Bull.* 65, *Amer. Rail. Engineer, etc., Association*) is in general use, in which the material (100 grms.) is distilled from an 8-ounce covered retort, the thermometer bulb being ½ inch above the surface of the oil, and distillation at the rate of 1 to 2 drops per second. The dimethyl sulphate test is used to distinguish between oil-products of benzene character or pyro-distillates (soluble), and paraffin or olefine hydrocarbons, produced by the distillation of mineral oil (insoluble in dimethyl sulphate). (See Sommer, *J. Ind. and Eng. Chem.*, 1910, **2**, 186.) A. R. T.

Purchase of Coal under Specifications. G. S. Pope. (*U.S. Geological Survey, Bulletin* **428**, 1-80.)—This paper gives in detail the results of Government purchases of coal for the fiscal year 1908-09, and the list of contracts with abstracts of specifications for the year 1909-10, and summarises the information on this subject obtained by the Survey. Since the publication of the last Bulletin on the subject (*U.S. Geological Survey*, No. 378), more accurate data have been accumulated as the result of experiments by the Survey staff and other chemists and engineers, as to the reduction in efficiency induced by high percentages of ash. As a result, the model specification now published provides for a bonus of 2 cents per ton for every 1 per cent. of ash below the contract standard (as against 1 cent per ton in earlier specifications) and, whilst tolerating 1 to 2 per cent. of ash above standard, imposes penalties for larger quantities of ash which penalties progress more rapidly than the ash percentage. For example, coal with ash 2 to 3 per cent., or 3 to 4 per cent., above standard is only penalised 2 and 4 cents per ton respectively, but 6 to 7 per cent. excess involves a penalty of 18 cents, whilst coal with higher ash content than this may be refused, or, at the option of the purchaser, accepted subject to substantial allowances of 25 to 35 cents per ton. These corrections in price to be paid are independent of and additional to the correction calculated from the ratio of calorific power of coal received to that guaranteed by vendor. In the past, the United States Government has purchased its anthracite on a specification, which simply provided heavy penalties for excessive ash. In future, tenders will be invited for the supply of anthracite on the same terms as apply to all contracts for bituminous coal—namely, payment at contract price multiplied by the ratio of the calorific power of the coal received to that guaranteed by vendor, ± a correction for ash content according to the scale published in this bulletin, and from which some typical figures have been given above. The bulletin gives useful information as to the award of contracts when inviting tenders on this "double basis," as it is called. Sellers in America are now so accustomed to this system that they usually, when offering coal, state the heat value and ash percentage which they are content to have made the basis of the contract, and this is preferred by the Government; but any tenderer may, if he prefers, submit a sample, which is then analysed in the Survey Laboratory, and becomes the basis of the contract, if one is entered into. G. C. J.

Deposits resulting from the Lubrication of Engines. J. E. Southcombe. (*J. Soc. Chem. Ind.*, 1911, 30, 261-264.)—The deposits from certain superheated steam cylinders were found to have the average composition Fe_3O_4, 86 per cent., organic matter 14 per cent. The organic matter included little or no free carbon, but consisted of solid bitumens containing 90 to 94 per cent. of carbon, and 6 to 9 per cent. of hydrogen. A brownish-black powder, formed in the lubricating pipes of forced feed engines using hydrocarbon oils, was found to consist of 54 per cent. unchanged oil (soluble in petrol), 22 per cent. bituminous matter soluble in ether, 12 per cent. bituminous matter insoluble in ether, but soluble in carbon bisulphide, 4 per cent. organic acids, 4 per cent. ash, and 4 per cent. water, etc. (by diff.). Only 87 per cent. of the ash was oxide of iron or copper. A black, gritty deposit, from the lubricating pipes of a steam turbine, gave ether-soluble matter (thick oil) 79 per cent., bituminous matter (soluble in carbon bisulphide) 9 per cent., organic acids 1 per cent., iron and iron oxide 4 per cent., cupric oxide 2 per cent., water, etc. (by diff.) 5 per cent. A black amorphous lumpy mass from the cylinder of a gas engine using producer gas contained tarry matter (soluble in ether) 43 per cent., insoluble bitumen and carbon 13 per cent., free sulphur (soluble in chloroform) 5 per cent., combined sulphur 4 per cent., and oxide of iron 35 per cent. G. C. J.

Characteristics of Oils from Papilionaceous Plants. C. Grimme. (*Chem. Rev. Fett- u. Harz-Ind.*, 1911, **18**, 53-55; 77-82.)—The oils, the characteristics of which are given below, were all obtained by extracting the seeds of the plants with ether. The oils from the seeds of the melilot and white melilot were drying oils, whilst the others were either non-drying or had only weak drying properties:

Oils.

Oil from—	Yield of Oil.	Sp. Gr.	Solidification Point.	Refractive Index.	Acid Value.	Saponification Value.	Iodine Value (Wijs).	Hehner Value.	Glycerol.	Unsaponifiable Matter.
	Per Cent.		° C.						Per Cent.	Per Cent.
Ornithopus sativus Brotero (bird's-foot).	8·96	0·918 (20° C.)	−17	1·4751 (20° C.)	19·4	185·3	69·0	93·5	9·07	1·84
Onobrychis satira (Turkish clover).	7·18	0·915 (25° C.)	−11	1·4770 (30° C.)	13·2	175·2	67·7	90·2	8·85	5·51
Lupinus albus (white lupin).	6·27	0·920 (20° C.)	−9	1·4742 (20° C.)	20·5	192·8	61·6	94·3	9·42	0·98
Lupinus angustifolius (blue lupin).	5·56	0·920 (20° C.)	−10	1·4725 (20° C.)	21·6	186·2	83·2	94·4	8·97	1·12
Lupinus luteus (yellow lupin).	5·41	0·920 (20° C.)	−5	1·4776 (20° C.)	16·5	185·0	68·3	94·7	9·21	1·14
Anthyllis vulneraria (wound-wort).	10·38	0·916 (25° C.)	−18	1·4756 (30° C)	13·1	189·0	71·6	92·9	9·61	2·36
Melilotus albus (white melilot).	6·63	0·931 (25° C.)	−10	1·4862 (30° C.)	15·5	187·9	71·4	93·25	9·42	2·05
Melilotus officinalis (melilot).	7·83	0·928 (25° C.)	−10	1·4760 (30° C.)	86·1	193·2	69·2	96·7	5·85	0·74
Medicago sativa (lucerne).	7·63	0·922 (25° C.)	−12	1·4766 (30° C.)	14·7	193·4	78·9	93·8	9·76	1·38
Trifolium incarnatum (pink clover).	6·18	0·910 (25° C.)	−9	1·4723 (30° C.)	21·4	181·3	61·6	94·1	8·73	1·57
Trifolium pretense (red clover).	14·78	0·914 (25° C.)	−14	1·4732 (30° C.)	8·3	191·8	64·1	93·1	10·02	1·93
Trifolium hybridum (bastard clover).	6·42	0·918 (25° C.)	−14	1·4757 (30° C.)	13·4	187·2	65·9	94·3	9·49	1·03
Trifolium repens (white clover).	6·81	0·910 (25° C.)	−16	1·4745 (30° C.)	10·5	189·4	68·5	93·3	9·77	1·77
Trifolium agrarium (yellow clover).	6·85	0·922 (25° C.)	−15	1·4757 (30° C·)	9·5	188·4	75·9	93·9	9·77	1·14
Trigonella fœnum græcum (fenugreek).	5·98	0·928 (25° C.)	−12	1·4738 (30° C.)	20·6	183·4	81·9	94·7	9·21	2·64
Luteus corniculatus (horn trefoil).	6·45	0·930 (25° C.)	−14	1·4729 (30° C.)	12·0	190·7	70·0	93·2	9·76	1·87
Galega officinalis (lagwort).	3·89	0·921 (25° C.)	−9	1·4728 (30° C.)	33·5	175·1	61·8	93·1	7·73	3·11

FATTY ACIDS.

Oil from—	Melting Point.	Solidification Point.	Refractive Index.	Neutralisation Value.	Iodine Value.	Mean Molecular Weight.
	°C.	°C.				
Ornithopus sativus Brotero	28·0	25·0	1·4593 (50° C.)	183·0	61·7	305·2
Onobrychis sativa Lam.	19·0	16·5	1·4574 (50° C.)	194·0	75·9	289·5
Lupinus albus	27·0	24·0	1·4559 (50° C.)	188·3	56·3	298·2
Lupinus angustifolius	24·5	23·0	1·4556 (50° C.)	187·2	67·6	300·2
Lupinus luteus	35·5	32·0	1·4600 (50° C.)	177·3	64·0	316·7
Anthyllis vulneraria	25·0	22·5	1·4640 (50° C.)	179·9	79·7	312·2
Melilotus albus, Desr.	27·0	23·0	1·4812 (50° C.)	178·5	74·8	314·6
Melilotus officinalis	38·0	35·0	1·4659 (50° C.)	169·3	64·7	331·9
Medicago sativa	32·0	30·0	1·4659 (40° C.)	183·4	77·8	306·2
Trifolium incarnatum	30·0	27·5	1·4620 (40° C.)	189·0	66·1	297·1
Trifolium pratense	33·0	29·5	1·4626 (40° C.)	185·0	66·0	303·6
Trifolium hybridum	28·0	24·0	1·4626 (40° C.)	189·0	69·5	297·1
Trifolium agrarium	32·0	28·0	1·4666 (40° C.)	185·3	78·3	303·2
Trigonella fœnum græcum	29·0	25·5	1·4626 (40° C.)	178·5	79·8	314·8
Lotus corniculatus	30·0	27·5	1·4620 (40° C.)	171·6	73·4	327·4
Galega officinalis	39·0	36·0	1·4672 (45° C.)	184·5	62·0	305·1

C. A. M.

Use of Dimethylaminobenzene-azo-benzene Sulphonic Acid as a Reagent in Microchemistry. M. E. Pozzi-Escot. (*Bull. Soc. Chim.* 1911 [iv.], 9, 22-26.)—Dimethylaminobenzene-azo-benzene sulphonic acid forms difficultly soluble metallic salts, some of which differ so much from the others in crystalline appearance as to be of use in microchemical analysis. Twenty-seven such salts are described and twenty figured in the text. It is suggested that the reagent affords a means of detecting small quantities of nickel in presence of much cobalt and *vice versâ*. Nickel gives rise to an amorphous yellowish-green precipitate, soluble in boiling water, from which, on cooling, it deposits pale yellow prisms, whose three dimensions are not very unequal. Cobalt, on the other hand, gives a crystalline precipitate at once, the crystals being violet or nearly black needles, the length of which may equal that of the prisms yielded by the nickel salt, but their other dimensions are very small indeed, being represented by single lines in the microphotographs accompanying the paper. G. C. J.

Influence of Potassium Permanganate upon Organic Bodies. J. Hetper. (*Anzeiger Akad. Wiss. Krakau*, 1910, Series A, 601-605; through *Chem. Zentralbl.*, 1911, 1, 927.)—The behaviour of organic compounds towards an acid solution of potassium permanganate depends upon their constitution, and it is therefore possible in this way to detect the presence of certain groups in the molecule. The method consists in heating a solution of the substance in 27 c.c. of water for one and a half hours at 92° to 93° C. with 25 c.c. of $\frac{N}{5}$-potassium permanganate solution containing 20 grms. of crystallised phosphoric acid per litre, then treating the hot liquid with 25 c.c. of $\frac{N}{5}$ oxalic acid solution containing 50 grms. of concentrated sulphuric acid per litre, and titrating the excess of the latter with $\frac{N}{5}$ permanganate solution. The following provisional conclusions were drawn by the author from results thus obtained: (1) Complete oxidation takes place in the case of all compounds soluble in water, alkali hydroxide solution, or phosphoric acid, provided they contain none of the groupings mentioned in (2), (3), and (4). In particular, all hydroxylated compounds such as polyvalent alcohols, phenols, etc., are completely oxidised. (2) Compounds with the constitution $CH_3.C\langle$, or $C_2H_5.C\langle$, etc., are not completely oxidised, since these radicles are oxidised into fatty acids, which undergo no further change. (3) Compounds containing the CH_3 group are oxidised with difficulty, and the more so in proportion to the nearness with which this group stands in relation to a COOH group, or a group oxidisable to COOH. The greater the distance between the two groups the more readily is the COOH group split up into CO_2 and H_2O, with the simultaneous oxidation into COOH of the carbon atom next to the disintegrated group. In this oxidation there is produced a homologous acid of the lower series. Acetic acid is exceedingly stable towards potassium permanganate, whilst propionic acid is less readily oxidised to acetic acid than is butyric acid to propionic acid. Compounds containing other hydrocarbon groups, such as C_6H_5, in the vicinity of the COOH group appear to behave in an analogous manner. (4) Dicarboxylic acids are completely oxidised when both COOH groups are in immediate contact (oxalic acid), or, at least, in combination with the same carbon atom (malonic acid). Other dicarboxylic acids (*e.g.*, succinic acid) are not oxidised. (5) Inorganic acid and basic groups are converted into the corresponding acids or bases (*e.g.*, NO_2 into HNO_3, NH_2 into NH_3, etc.). For purposes of comparison the terms *molar* and *gram values* are suggested. The molar value is the number of c.c. of N-potassium permanganate solution required to oxidise 1 grm. molecule of the substance; whilst the gram value is the quantity (in c.c.) required to oxidise 1 grm. C. A. M.

Estimation of the Activity of Oxidising Agents by Means of the Quinonoid Imonium Salts of Benzidine. W. Madelung. (*Ber.*, 1911, **44**, 626-631.)—If any oxidising agent be added to an excess of an aqueous solution of benzidine, care being taken to maintain a neutral reaction, a precipitate of cornflower-blue microscopic needles is formed, which is sparingly soluble, and in presence of small quantities of mineral salts is perfectly insoluble. This compound is an analogue of the *meri*-quinonoid salts of diphenoquinone diimine described by Schlenk, the negative radical depending on the particular oxidising agent employed. The formation of the blue oxidation product of benzidine may be applied for analytical purposes, since it retains in the active form the whole of the oxidising power of the active oxygen employed in its formation, particularly in physiological investigations, and in estimating the oxidising power of coloured solutions, which cannot be titrated direct. The benzidine derivative may be precipitated, isolated, and caused to liberate iodine from acidified potassium iodide, which is then titrated with standard thiosulphate. If the blue compound be produced in neutral aqueous solution and collected, the blue colour is immediately discharged on the addition of hydrochloric acid; there remain brown flakes of the dichlorimide, which dissolve when potassium iodide is added as a preliminary to titration. The final result is the same as if the oxidising agent had been titrated directly, but in certain cases the precipitation and separation of the oxidising function presents distinct advantages. In the investigation of blood, and in the study of the activification of hydrogen peroxide by peroxydases, the author has used the method with considerable success. Sodium chloride is employed to increase the insolubility of the precipitate. J. F. B.

Separate Estimation of Potassium Bitartrate and Calcium Tartrate. P. Carles. (*Bull. Soc. Chim.*, 1911, [iv.], **9**, 199-202.)—This is said to be important both to those who extract the marc only with boiling water, thus losing most of the calcium tartrate, and to those who treat the marc with dilute sulphuric acid to recover the whole of the tartaric acid. When working for the recovery of the whole of the tartaric acid in material in which the calcium salt preponderates, it is preferable to recover the whole of the acid in the form of calcium salt.

The marc (200 grms.) is mixed with 200 c.c. of water in which 2·5 grms. of anhydrous sodium carbonate have been dissolved. If necessary, more water is added to cover the sample. The dark-coloured liquid is poured off as far as possible into another vessel and then back on to the marc, and this process is repeated once or twice. At the end of an hour or two the liquid is again decanted. It is now quite pale and neutral or faintly acid to litmus. A further 1 grm. of sodium carbonate (or an equivalent amount of a concentrated solution) is added to it, when it regains its dark colour. It is again poured over the marc and, at intervals of about two hours, is decanted, and, whenever it is found to be substantially decolorised, its alkalinity and colour is to be restored by a further addition of 1 grm. (or 0·5 grm.) of sodium carbonate. This process is continued for forty-eight hours, and about 6 to 7 grms. in all of sodium carbonate is usually required. At the end of the period the liquid should be pale but distinctly alkaline to litmus, and should maintain its alkaline reaction for at least five hours. It is then finally decanted, and the marc allowed to drain for a quarter of an hour. The marc is next covered with distilled water, which is decanted off after two hours, and this washing by decantation repeated twice more. The combined extracts, which contain as Rochelle salt all the tartaric acid originally present as potassium bitartrate, are nearly, but not quite, neutralised with acetic acid, and are concentrated at a temperature below boiling to about 125 c.c. If much pectinous matter separates, resort must be had to filtration; but the extraction of the marc by liquor, which is never strongly alkaline, is designed to reduce the extraction of such matters to a minimum. When the solution has been concentrated to 125 c.c., acetic acid is added until the reaction is distinctly acid, and this is followed by 10 c.c. of a saturated solution of calcium acetate. The mixture is maintained at a temperature near its boiling-point, and, at five-minute intervals, further 10 c.c. portions of calcium acetate are added up to 70 to 80 c.c. in all. Calcium tartrate begins to crystallise at the first addition, and after the last the beaker is set aside for twenty hours. At the end of that time the crystals and mother liquor are transferred to a 250 to 300 c.c. basin, an equal bulk of cold water added, and the liquor decanted into a second basin, and, if it carries any crystals with it, it is decanted from them into a third. Small quantities of pectinous matters and other impurities are thus easily eliminated. The crystals are washed three times with cold water by decantation in the same way, and are then drained, dried, and weighed. The weight × 0·723 gives the weight of potassium bitartrate in 200 grms. of the marc.

The extracted marc is covered with water, and the water decanted off, mixed with 5 c.c. of hydrochloric acid, and again poured over the marc. The acid liquor is decanted off and returned to the beaker twice an hour for three or four hours. At the end of that time it is decanted off and replaced by water, and after an hour this water is decanted off. The marc is washed twice more in a similar manner by decantation, and the united acid liquid and washings are then concentrated to 125 c.c. In this solution, which contains all the tartaric acid originally present as calcium tartrate, the tartaric acid is precipitated as calcium tartrate by addition of a saturated solution of calcium acetate as already described. G. C. J.

Colour Reaction of Proteins with Sodium Nitroprusside. V. Arnold. (*Zeitsch. physiol. Chem.*, 1911, **70**, 300-309.)—A number of proteins, particularly the products obtained by the action of pepsin on albumin or muscular fibre in the presence of hydrochloric acid, yield an intense purple coloration when a small quantity of their aqueous solution is treated with a few drops of a 5 per cent. sodium nitroprusside solution, and the mixture then rendered ammoniacal. The coloration, which is destroyed by acetic acid, is relatively stable, and is produced by the cystein group in the protein molecule; it is not due to sulphur compounds split off from the protein. Egg-albumin does not yield the reaction until after it has been subjected to peptic digestion, but the soluble proteins obtained from the crystalline lens of the eye give an intense coloration, as do also aqueous extract of many of the other organs of the body. Vegetable seeds frequently contain the substance which gives the reaction, but the coloration obtained is always less intense than in the case of animal substances. W. P. S.

Notes on Hubener's Caoutchouc Tetrabromide. R. Becker. (*Gummi Zeit.*, 1911, 25, 531.)—Gutta, and probably balata, yield bromine compounds similar to those obtained in the case of caoutchouc, and Hubener's method (ANALYST, 1909, **34**, 355) is, therefore, not suitable for the separation of gutta from caoutchouc.

Estimation of "Mineral Rubber" and Similar Products in Rubber Articles. R. Becker. (*Gummi Zeit.*, 1911, **25**, 598.)—Carbon bisulphide extracts "mineral rubber," asphaltum, etc., but does not appreciably dissolve rubber.

Estimation of Iodine in Alcoholic Solutions. G. Favrel. (*Ann. Chim. anal.*, 1911, **16**, 12-13.)—Desvigne's process (weighing as silver iodide after conversion into potassium iodide) and the volumetric process suggested by Libeau (titration with silver nitrate after treatment with zinc powder) give the same result as titration with thiosulphate.

Anhydride of Lactic Acid. A. A. Besson. (*Chem. Zeit.*, 1911, **35**, 26.)—Lactic acid frequently contains a quantity of the anhydride; and the total acidity of the substance (acid *plus* anhydride) is only obtained when the sample, after neutralisation of the acid, is treated with an excess of alkali, allowed to stand for ten minutes, then acidified and titrated.

INORGANIC ANALYSIS.

Estimation of Free Acid and Basic Alumina in Aluminium Salts. T. J. I. Craig. (*J. Soc. Chem. Ind.*, 1911, **30**, 184-185.)—The method is based on the fact that an excess of neutral potassium fluoride decomposes aluminium salts, forming two stable compounds neutral to phenolphthaleïn, while any free acid remains unaltered, thus:

$$Al_2(SO_4)_3 + (xH_2SO_4) + 12KF = 2(AlF_3,3KF) + 3K_2SO_4 + (xH_2SO_4).$$

The precipitated aluminium potassium fluoride is insoluble in presence of a moderate excess of potassium fluoride, and is not appreciably attacked by dilute acid or dilute caustic alkali.

Pure potassium fluoride is dissolved in water, and the solution brought to a sp. gr. of 1·45. It is neutralised, if necessary, with caustic potash or sulphuric (or hydrofluoric) acid till about 1 c.c. in 10 c.c. water shows a faint pink colour with phenolphthaleïn. Insoluble matter is filtered off, and the filtrate, without washing the paper, is diluted to sp. gr. 1·35, containing about 0·5 grm. potassium fluoride per c.c. The reagent is stored in a glass bottle coated internally with wax. Standard sulphuric acid and caustic potash must be free from alumina and similar bases, and the acid is standardised against anhydrous sodium carbonate (using methyl orange), the alkali then being run against the acid in about 40 c.c. of water, to which 10 c.c. of the above potassium fluoride solution has been added, phenolphthaleïn being the indicator (0·2 per cent. solution).

The substance to be examined is dissolved in water, so that 1 to 3 grms. alumina are present in 200 c.c. The solution is filtered from insoluble matters, and 20 c.c. gradually added, with stirring, to a mixture of 10 c.c. of potassium fluoride solution, 50 to 60 c.c. water, and 0·5 c.c. phenolphthaleïn solution. If neutral, the liquid will be slightly pink, and one drop of $\frac{N}{2}$ alkali will intensify the colour. If free acid be present, standard alkali is run in till a faint permanent pink is produced. If the substance is basic, caustic alkali will be liberated:

$$Al_2O(SO_4)_2 + 12KF + H_2O = 2(AlF_3.3KF) + 2K_2SO_4 + 2KOH.$$

This alkalinity cannot be directly titrated, however, as a portion is precipitated as a basic double fluoride neutral to phenolphthaleïn. Standard acid is therefore added till the pink colour disappears, and the amount required noted. A similar amount of standard acid, plus a known excess equal to 4 c.c. $\frac{N}{2}$ acid (more if necessary), is then added to a fresh portion of the filtered solution of the basic substance, the liquid heated to cause the basic alumina and the acid to react, and, after cooling, the method carried out as before by addition to the fluoride solution, the amount of free acid shown being noted. The amount of added acid which has disappeared may thus be calculated in terms of basic alumina (1 c.c. $\frac{N}{2}$ acid = 0·0085 grm. Al_2O_3). The volume of liquid after titration may be 60 to 120 c.c. In presence of sodium salts, 20 to 30 c.c. of neutral saturated solution of potassium sulphate are added to the liquid, before it is mixed with the fluoride, to minimise the tendency to form basic sodium double fluoride. Ammonia, if present, must be removed by heating with a known excess of standard alkali before mixing with the fluoride, more of which latter solution is required in the presence of iron salts in the material under examination. If the almost colourless iron potassium fluoride precipitate darkens during the titration, owing to decomposition by the alkali, too little fluoride is present. The amount of free acid in iron and zinc salts may be determined by this method, but larger amounts of fluoride solution must be used. A. R. T.

Estimation of Arsenic in Insecticides. E. B. Holland. (*J. Ind.* and *Eng. Chem.*, 1911, 3, 168-171.)—The author recommends the following method for the estimation of arsenic in arsenical insecticides, including substances, like Paris green, containing copper: 2 grms. of the finely-powdered sample are boiled for five minutes with 50 c.c. of sodium acetate solution (1 : 2). The liquid is cooled, 60 c.c. of diluted (1 : 3) hydrochloric acid added, and the mixture shaken to complete solution, when the liquid is diluted to 500 c.c. and filtered. Twenty-five or 50 c.c. of the solution are neutralised with sodium bicarbonate, 25 c.c. of sodium potassium tartrate solution (10 per cent.) added (in the case of copper arsenites only) to dissolve precipitated copper, and after adding 3 grms. of sodium bicarbonate, and making the volume up to 100 c.c., the solution is titrated with $\frac{N}{20}$ iodine solution in presence of 2 c.c. of starch-paste (1 : 200) till a permanent blue colour is obtained. Towards the end of the reaction the flask should be corked and shaken vigorously. The original residue is weighed as insoluble matter after ignition. The presence of cuprous and ferrous compounds, sulphurous and nitrous acids, or other oxidisable substances, is a source of error in the iodine titration method.

Iodine Method for Arsenates.—Two grms. of the finely-ground sample (*e.g.*, lead arsenate) are boiled with 60 c.c. of diluted nitric acid (1 : 3), the liquid diluted to 500 c.c., and filtered. Fifty or 100 c.c. are evaporated with 10 c.c. of sulphuric acid (2 : 1), the residue heated at 150° to 200° C. in an air-bath to expel moisture, and finally heated on a plate till dense white fumes of sulphuric acid are given off, and all nitric acid is expelled. A little water is added, and the solution filtered under diminished pressure, and the filter washed till a volume of 100 c.c. is obtained. Ten c.c. of potassium iodide solution (16·5 per cent.) are added, and the liquid boiled till the free iodine is expelled, when the solution will become almost colourless. The liquid is diluted, cooled immediately, neutralised with sodium hydroxide, and acidified slightly with sulphuric acid. Starch-paste is added, and if free iodine is present, $\frac{N}{20}$ thiosulphate solution is added carefully, with vigorous shaking, till the blue colour disappears. Excess of sodium bicarbonate is next added, and the titration with $\frac{N}{20}$ iodine carried out as usual, calculating as arsenic oxide. Insoluble matter in the original liquid is weighed as before. Practically no difference was noted in the titration when the lead sulphate was not removed. A. R. T.

Estimation of Ash in Vegetable and Animal Substances. E. Fleurent and L. Lévi. (*Comptes rend.*, 1911, **152**, 715-718.)—The loss of phosphoric acid varies very greatly in estimating the ash by ignition either in the open air or by Schlösing's method (ignition in a current of carbon dioxide followed by a slow current of oxygen). Thus, in the authors' estimations it ranged from 7·31 per cent.

in the case of white haricots, to 49 per cent. of the total quantity of phosphorus in the case of two varieties of barley. The causes of the loss are the action of carbon and of silica upon the acid phosphates in the ash, and also the removal of part of the phosphorus in the form of a volatile organic compound, this being particularly noticeable in the case of substances rich in fat. In order to obtain an accurate estimation of the ash, the authors first extract the fat, when present in large proportion, and carbonise the residue at as low a temperature as possible. The residual mass is then pulverised and mixed with a solution (or cream) of calcium oxide, containing 0·04 grm. to 0·150 grm. of CaO per 10 grms. of the organic material according to its richness in phosphoric acid, and the ignition completed by Schlösing's method. Finally, the amount of added calcium oxide is deducted from the weight. In this way a perfectly white ash is obtained without the losses inevitable by the other methods. Thus, in the analysis of three samples of wheat, the amounts of ash obtained by direct ignition were 15·41, 17·68, and 18·84 per cent., as against 16·92, 19·19, and 20·34 per cent. respectively by the new method. C. A. M.

Colorimetric Estimation of Minute Quantities of Chromium. P. Koenig. (*Chem. Zeit.*, 1911, 35, 277-278.)—The trace of chromium—for example, that occurring in the ash of plants grown in certain soils—is obtained in solution as chromate, and the solution mixed with a solution of disodium, 1·8-dihydroxynaphthalene, 3·6-sulphonate, which develops a distinct and permanent cherry-red coloration when even so little as 0·0008 mgrm. of chromium, as chromate, is present in 10 c.c. of the solution. The solution must not be alkaline, as the reagent gives a pink coloration with alkalis, but it may be made faintly but distinctly acid with any mineral acid, and the addition of a few c.c. of phosphoric acid is recommended in every case, as this addition inhibits the formation of coloured compounds of iron, tungsten, and uranium, which might otherwise interfere. To 10 c.c. of the solution to be tested 1 c.c. of a 1 per cent. solution of the reagent is added, together with a few c.c. of phosphoric acid. The intensity of the coloration developed is compared with that produced by a standard solution of potassium bichromate. The comparison is most easily made when the solutions to be compared contain from 0·001 to 0·01 mgrm. of chromium. Stronger solutions give rise to violet-red colours, which are less easily compared. G. C. J.

New Reaction for the Differentiation of Cobalt and Nickel. H. Weil. (*Bull. Soc. Chim.*, 1911, [iv], 9, 20-22.)—The reaction depends on the formation in neutral solution of precipitates of basic chromate of nickel or cobalt. If a sufficient quantity of a solution of potassium chromate be added to a neutral solution of a cobalt salt, a red-brown precipitate of $CoCrO_4.CoO.Aq$. forms. If the concentration of cobalt exceed 2 grms. per litre, the precipitate appears in the cold within a few seconds. Even very dilute solutions yield a visible precipitate on boiling. The precipitate adheres firmly to the glass vessel.

Nickel gives rise to a chocolate-brown precipitate, but this appears very slowly in the cold, even in concentrated solution. On heating to boiling, however, $NiCrO_4.2NiO$ separates, and is distinguished from the corresponding cobalt compound, not only by its colour, but by the fact that it shows little tendency to form on the sides of the glass vessel. Quantities of cobalt or nickel so small as 0·03 mgrm. may be detected in this way.

When occurring together and both in quantity, cobalt may be detected by the appearance of the precipitate in the cold, and nickel by boiling the filtrate from the cobalt precipitate. Small quantities of nickel in presence of much cobalt may be detected as follows: The filtrate from the cobalt precipitate is boiled, and if the resulting precipitate contains enough cobalt to mask any small amount of nickel, it is dissolved in ammonia and the solution boiled to expel the ammonia. If cobalt only is present, the resulting precipitate will be a dirty green, but a very small amount of nickel will give rise to the characteristic chocolate-brown colour. Nickel can be thus detected in presence of 100 times its weight of cobalt. One part of cobalt in presence of 50 parts of nickel can be detected in the same way. Nickel holds up small quantities of cobalt in the cold, but on boiling both come down. On dissolving in ammonia and reprecipitating by evaporation, if the cobalt-nickel ratio equals or exceeds 1 : 50, the precipitate will have a distinct green tinge. G. C. J.

Separation of Tantalum and Columbium. H. W. Foote and R. W. Langley. (*Chem. News*, 1911, **103**, 103-104.)—The authors have experimented with the method of Weiss and Landecker (*Zeitsch. anorg. Chem.*, 1909, **64**, 65), which consists essentially in fusing the mixed oxides of tantalum and columbium with sodium carbonate, containing a little nitrate, and treating with hot water. A portion of the tantalum remains as insoluble sodium tantalate, and may be filtered off and treated separately. That remaining in solution is precipitated by a current of carbon dioxide, when the tantalum is stated to be completely removed, leaving columbium in solution. Foote and Langley's experience is in harmony with that of Rose and of Ostwald, that both elements are almost completely precipitated by carbon dioxide under the above conditions. Variations in the amounts of reagents used and of the concentration of the solutions yielded no better results. A. R. T.

Indirect Method for determining Columbium and Tantalum. H. W. Foote and R. W. Langley. (*Chem. News*, 1911, **103**, 52-55.)—Penfield and Ford (*Am. J. of Sci.*, 1906, **22**, 61) proposed to calculate the composition of any mixture of columbic and tantalic oxide from its sp. gr. and that of pure columbic and tantalic oxide, on the assumption that the sp. gr. of mixtures was a linear function of the composition. This assumption is shown to be erroneous by the authors, who have determined the sp. gr. of a number of mixtures of known composition; but these experimental results now make it possible to calculate the composition of mixtures of the two oxides. It is important that the oxides be prepared by a uniform process, as the manner of preparation is not without effect on the sp. gr. The mixed oxides as separated in the course of analysis are fused with 6 parts of acid potassium fluoride, the fusion is dissolved in 200 c.c. of hot water containing a little hydrogen fluoride, the solution made alkaline with ammonia, and the precipitate filtered off on a rubber funnel, and washed with ammoniacal water. The precipitate is redissolved in a platinum dish, using as little hydrogen fluoride and water as possible, and the solution evaporated to dryness. Sulphuric acid (10 c.c.) is added to the residue, and the liquid heated to expel hydrogen fluoride, after which 200 c.c. of water and excess of ammonia are added, and the precipitate filtered off and washed with dilute ammonia. Finally, the precipitate is ignited for an hour over a blast-lamp, ground to powder with water, and its sp. gr. determined as follows: The unweighed material and water is transferred to a beaker and boiled hard for thirty minutes by immersing in the beaker a coil through which an electric current passes. As much as possible of the solid material is transferred to a small pycnometer, which is filled with water and weighed after adjustment of the temperature. The solid material is then washed out of the bottle into a platinum dish, the wash-water boiled away, and the residue ignited and weighed. The following results are given:

Composition, Per Cent. Ta_2O_5.	Sp. Gr.	Composition, Per Cent. Ta_2O_5.	Sp. Gr.
0	4·552	65	6·737
5	4·650	70	7·083
10	4·746	75	7·441
20	4·929	80	7·654
30	5·200	85	7·961
40	5·474	90	8·098
50	5·850	95	8·243
60	6·434	100	8·716

Duplicate analyses of minerals calculated by interpolation from the above table do not differ by more than 0·2 per cent. When 1 grm. or more of mixed oxide is available, the results are within 1 per cent. of those yielded by Marignac's method, which is not only tedious, but probably less accurate than this indirect method.
G. C. J.

Pot Assay of Gold and Silver in Ores containing much Copper. **J. Loevy.** (*Chem. Zeit.*, 1911, **35**, 278.)—The necessity for repeated scorification when determining gold and silver in highly cupreous materials may be avoided as follows: If the ore contains less than 12 per cent. of copper, 50 grms. are mixed with 50 grms. of sand, and the mixture divided into two portions, each of which is subjected to ordinary pot assay in a G-size crucible with suitable proportions of sodium carbonate, litharge, borax, argol, and a salt cover. Sufficient argol should be taken to yield a button weighing 30 to 40 grms. from each assay. These two buttons are scorified down together until a button weighing 30 to 35 grms. is obtained. This button will be so far free from copper that it may be cupelled without further scorification. In the case of ores containing 24 per cent. of copper, a mixture of 50 grms. ore and 150 grms. sand is distributed among four G-size crucibles or two J-size crucibles. The resulting buttons are scorified down together until a button weighing 30 to 35 grms. is obtained, and this is then cupelled.
G. C. J.

Estimation of Bromine in Presence of Chlorides and Iodides. P. Clausmann. (*Bull. Soc. Chim.*, 1911 [iv.], **9**, 188-192.)—Distillation with a strongly acid solution of potassium bichromate serves well enough for the estimation of notable quantities of bromine, but for the estimation of traces in presence of much chloride it is unsatisfactory, as prolonged distillation is necessary to carry over all the bromine, and water must be added from time to time, as at high concentrations chromyl chloride passes over. It has been proposed to effect the oxidation in a closed vessel, and subsequently to extract the cooled mixture with carbon bisulphide, to wash the carbon bisulphide solution, and then to mix it with iodide of potassium and sodium bicarbonate, and to titrate the liberated iodine. In the author's hands this method gives low results, the error increasing with the amount of bromine to be estimated. Accurate results may be obtained as follows: The saline solution to be examined is concentrated to a few c.c. and placed in a 200 c.c. flask, of which the upper part of the neck has been drawn out to a narrow tube about 6 inches long. From 0·5 to 1 grm. of chromic acid and 2 to 4 c.c. of sulphuric acid are added, and enough water to bring the contents of the flask up to about 100 c.c. The narrow neck of the flask is given a right-angled bend and the end drawn out to a point, easily breakable, and sealed. The flask, with its contents, is then heated for an hour in a boiling brine bath. It is then cooled, and the neck fitted, by means of a rubber stopper, to a condenser. The other end of the condenser carries a bend which dips into an acidulated solution of potassium iodide. The point of the drawn-out neck of the flask is next broken by pressure against the condenser tube, and the contents of the flask heated by a flame. The whole of the bromine (and iodine if present) passes over with the first 50 c.c. of distillate, the concentration of acid in the flask remaining much too low to cause any danger of the formation of chromyl chloride. The liberated iodine is determined by means of thiosulphate and calculated to bromine, after deducting any iodine which a separate estimation may have shown to be present in the sample.

G. C. J.

Estimation of Small Quantities of Iodides in the Presence of other Compounds. R. Bernier and G. Péron. (*J. Pharm. Chim.*, 1911, **3**, 242-248.)—The following modification of the oxidation method of Longi and Bonavia (*Gazz. Chim. Ital.*, 1898, **28**, 325) is claimed to be as accurate as the gravimetric method, and to estimate with accuracy as little as 0·0002 grm. of iodine. A quantity of the solution of the iodide (containing from 0·01 to 0·03 grm. of combined iodine) is diluted to about 50 c.c., neutralised if acid, and then rendered faintly alkaline with sodium hydroxide solution. A few crystals of potassium permanganate, so as to give a slight excess, are now introduced, and the liquid boiled for a few minutes, after which the excess of permanganate is reduced by the addition of about 5 c.c. of alcohol, and the liquid cooled, made up to 110 c.c., and filtered. One hundred c.c. of the filtrate are collected, and acidified with 10 c.c. of acetic acid, 5 c.c. of a 10 per cent. solution of potassium iodide added, and the liberated iodine titrated with $\frac{N}{10}$ thiosulphate solution. The number of c.c. used, plus one-tenth thereof, multiplied by 0·0127 and divided by 6, gives the weight of iodine in the quantity of solution taken. In the case of traces of iodine (below 0·002 to 0·004 grm.) a $\frac{N}{100}$ solution of thiosulphate is used for the titration. The method gives accurate results in the presence of chlorides, bromides, and fluorides, and also of chlorates and bromates, after previous reduction by means of sodium bisulphite. It can also be used in the presence of substances that readily react with iodine (sulphides, sulphites, thiosulphates, etc., arsenites and antimonites). In the case of cyanides the liquid must be boiled with acetic acid to eliminate the hydrocyanic acid. Ammonium salts must also be decomposed by boiling the liquid with alkali before estimating the iodide.

C. A. M.

Separation of Lime from Large Quantities of Magnesia. E. Murrmann. (*Monatsh.*, 1911, **32**, 105-115.)—When the magnesia-lime ration exceeds 50 : 1, the only satisfactory method is that previously described by the author (ANALYST, 1910, **35**, 455). When the magnesia-lime ratio is less than this, the following simple method gives good results: To the neutral solution, which may contain as much as 0·4 grm. magnesia per 100 c.c., 3 c.c. of strong ammonium chloride solution and 3 drops of hydrochloric acid are added, and the mixture heated to boiling. Concentrated oxalic acid solution and 3 or 4 c.c. of quinoline are then added, and after ten minutes the precipitated calcium oxalate may be filtered off. If it is required to estimate magnesium in the same portion of the original solution, quinoline cannot be used, but pyridine may be substituted for the quinoline, and subsequently expelled from the filtrate. When using pyridine, the reaction of the solution needs to be more exactly adjusted by means of methyl orange. The pyridine should be added as a 2 per cent. solution, drop by drop, and the addition must cease before the red colour of the methyl orange gives place to yellow. When using pyridine, the results are slightly improved by cold precipitation and fifteen hours' standing before filtration. The magnesia in the filtrate can be estimated, as usual, after one or two evaporations with ammonia to expel the pyridine. Large quantities of salts of the fixed alkalis lead to lime being overestimated by either of these methods. For example, 3 grms. of alkali chloride or sulphate per 100 c.c. may lead to 0·05 grm. lime being over-estimated by 6 or 7 per cent.; but even in such cases solution of the precipitate in acid and reprecipitation by ammonia and ammonium oxalate will give exact results.

G. C. J.

Differentiation of Minerals by Means of Colour Reactions. P. Gaubert. (*Bull. Soc. Franç. Minéral*, 1910, **33**, 324-326; through *Chem. Zentralbl.*, 1911, **1**, 684-685.)—On adding to the finely-powdered mineral a solution of morphine, naphthol, or other substances in sulphuric acid, a reaction, the appearance of which is sometimes accelerated by the addition of water, is obtained after some time. In addition to the compounds employed by Levy (*Comptes rend.*, 1886, **103**, 1074, 1195) for the identification of metallic acids, a sulphuric acid solution of papaverine may be employed. This gives a red coloration, changing to blue in the case of vanadic, tungstic, chromic, and titanic acids, and to brown with stannic acid. In this way it is possible to distinguish, by means of the colour, between cassiterite and rutile, and, by the speed of coloration, between wulfenite (very rapid) and vanadinite and crocoite (fairly slow). Rutile, brookite, anatase, ilmenite, titanite, and perowskite give, in the finely-powdered condition, a carmine red coloration with morphine. If the test is made with equal quantities of the substance, sulphuric acid and morphine, different periods of time will elapse before the powder assumes a red colour. Thus amorphous titanic acid reacts very rapidly, whilst rutile is coloured less readily than brookite, which in turn offers greater resistance than anatase, although it is not easy to distinguish between the two latter minerals. On the addition of a suitable proportion of water a rose coloration is immediately obtained with all three minerals. Whilst keilhanite shows the reaction within two hours, the colour does not appear with titanite until after twenty-four hours. In the case of perowskite the coloration is produced in two to three hours, or immediately after the addition of water. Minerals containing rare earths will show the reaction after an hour, and without any preliminary treatment with a solvent. Wolframite is less readily acted upon than the other minerals mentioned. The colour reaction with cassiterite is readily produced, and affords a means of distinguishing that mineral from zirconium compounds. C. A. M.

Analysis of Molybdenum Glance. W. Trautmann. (*Zeitsch. angew. Chem.*, 1911, **24**, 207.)—Molybdenum glance of Norwegian origin is now appearing on the market containing 5 to 14 per cent. of magnesia (usually about 6 per cent.), 1 to 5 per cent. of copper as copper pyrites, and 0·3 to 4·0 per cent. of zinc as blende. When attempt is made to estimate the molybdenum in such ores by Gilbert's method (*cf.* Lunge's *Technical Methods*), nearly all the zinc, copper, and magnesium get weighed as MoO_3. The author's method (*Chem. Zeit.*, 1909, **33**, 1106; ANALYST, 1910, **35**, 547) is unaffected by the presence of copper and magnesium, but zinc interferes. In presence of zinc, the molybdenum solution must be boiled with ammonium polysulphide, the zinc sulphide filtered off, and the molybdenum in the filtrate precipitated by addition of acid. The MoO_3 finally obtained must, after weighing, be purified as follows, as it may contain nearly 1 per cent. of impurities: It is fused with potassium bisulphate, the melt dissolved in hot ammonium carbonate solution, and any alumina and silica filtered off and washed with an ammoniacal solution of ammonium nitrate, dried, ignited, and weighed, and its weight deducted from that of the impure WO_3 previously weighed (*cf.* ANALYST, 1909, **34**, 540).

G. C. J.

Estimation of Nitrogen in Nitrates, Gun-Cotton, etc. M. Marqueyrol and D. Florentin. (*Bull. Soc. Chim.*, 1911 [iv.], 9, 231-240.)—Having found differences between the results yielded by Schloesing's and Lunge's methods for the estimation of nitrogen in nitrates, etc., the authors carried out an investigation with the object of discovering the source of the errors. In the case of Schloesing's method (decomposition of the nitrate by boiling with ferrous sulphate and hydrochloric acid, and measuring the nitric oxide formed) it was found that the carbon dioxide, employed for filling the apparatus before the mixture in the flask is boiled, is never quite pure, and always leaves a small residue of gas which is not absorbed by potassium hydroxide; secondly, the hydrochloric acid used evolves a trace of dissolved air when heated; and, thirdly, the nitric oxide reacts with the potassium hydroxide with the formation of potassium nitrite, nitrous oxide, and nitrogen. The first two errors increase the result obtained by the method, whilst the third decreases it, and, in practice, the errors compensate each other if the total time taken for the operation does not exceed twenty-five minutes, and the nitric oxide collected over the potassium hydroxide solution is not shaken with the latter for more than five minutes. The agitation of the gas must, however, be thorough, or another cause of error may be introduced—namely, incomplete absorption of the carbon dioxide. Lunge's nitrometer method gave accurate results, provided that the strength of the sulphuric acid employed was not more than 94 per cent. When a more concentrated acid is used, a portion of the nitric acid is decomposed, and the results obtained are too low.

W. P. S.

Study of the Phenolsulphonic Acid Method for the Estimation of Nitrates in Water. III. Chief Sources of Error. E. M. Chamot, D. S. Pratt, and H. W. Redfield. (*J. Amer. Chem. Soc.*, 1911, **33**, 366-381.)—Continuation of the work of Chamot and Pratt (Analyst, 1909, **34**, 456; 1910, **35**, 321). The authors refer to most of the recent criticisms of the method, notably to those of Farcy (Analyst, 1910, **35**, 81) and of Perrier and Farcy (Analyst, 1909, **34**, 174), and describe experiments made with the object of determining all the sources of error, and so paving the way to a method which shall, as far as possible, avoid all of these (*cf.* next abstract). The principal conclusions reached are as follows: Sulphonic acid reagents containing mono-acids are subject to change with age, and yield results greatly affected by temperature, concentration, variation in character of alkali, etc. They cannot be satisfactorily employed with permanent standards. In order that accurate results may be obtained, chlorides, carbonates, and organic matter must be removed. Nitrites present in small amount (less than 1 part per million) do not appreciably affect the results, but when exceptionally high must be destroyed or corrected for. A sulphonic acid of definite composition should be used (*cf.* next abstract).

G. C. J.

Study of the Phenolsulphonic Acid Method for the Estimation of Nitrates in Water. IV. A Modified Method. E. M. Chamot, D. S. Pratt, and H. W. Redfield. (*J. Amer. Chem. Soc.*, 1911, **33**, 381-384.)—The reagent is prepared by dissolving 25 grms. phenol in 150 c.c. sulphuric acid, adding 75 c.c. of fuming sulphuric acid (13 per cent. SO_3) and heating for two hours at about 100° C. The reagent thus prepared contains no mono-acids and no tri-acids, and may be heated with water residues for hours without any development of interfering colours. It is also far less sensitive to the interfering action of substances causing errors than any of the reagents hitherto described, which all contain notable amounts of mono-sulphonic acid. Before proceeding to estimate the nitrates in a water, its alkalinity and chlorine and nitrite content are determined. If nitrites are present in amount exceeding 1 part per million, they are oxidised to nitrate by means of hydrogen peroxide and a correction applied to the amount of nitrate found. Smaller amounts of nitrite may be neglected. To 100 c.c. of the sample (or less, if the nitrate nitrogen is believed to exceed 10 parts per million) sufficient dilute standard sulphuric acid is

added to almost, but not quite, neutralise the alkalinity, and then a standard solution of silver sulphate almost, but not quite, sufficient to combine with all the chlorine. The mixture is boiled and, after addition of alumina cream, is filtered and the precipitate washed with hot water. The filtrates and washings are evaporated to dryness, 2 c.c. of the disulphonic acid reagent added, and the mixture rubbed with a rod to ensure intimate contact. If much iron or magnesia be present, the residue will appear compact and vitreous, and in such cases warming on the water-bath is advisable. After diluting with water, caustic potash solution is added slowly until the maximum colour is developed. Not much more than 1 grm. of potash is usually necessary, and, provided enough is added, the use of a large excess (*e.g.*, 5 grms.) is without influence on the results. The colour comparison is made with a standard solution of tripotassium nitrophenol disulphonate. This is made as follows: To the phenoldisulphonic acid reagent, powdered potassium nitrate is added in very small quantities at a time, until 0·1076 grm. has been added for each c.c. of reagent taken, stirring after each addition. The product is diluted and treated with dry barium carbonate until a deep yellow colour results. The mixture is filtered and the precipitate washed well with boiling water, to extract the barium nitrophenoldisulphonate. Potassium carbonate is added to the filtrate and washings until these are alkaline, when the solution is filtered and concentrated to recrystallisation. The product is purified by recrystallisation, but no attempt is made to obtain an absolutely pure product, as this involves drying in a current of carbon dioxide. A solution of the crystals is made up and standardised against a known weight of pure potassium nitrate treated with the reagent in the usual manner. The standard solution remains unaltered when exposed for many months to diffused daylight in clear glass bottles.
G. C. J.

Estimation of Dissolved Oxygen. L. W. Winkler. (*Zeitsch. angew. Chem.*, 1911, 24, 341-343.)—The method depends on the colour produced when chloro-quinol (adurol) is dissolved in water containing dissolved oxygen, and a trace of ammonia added. As ammonia may give rise to a precipitate with a magnesian water, an ammoniacal solution of ammonium bromide is used, the bromide being preferred to the chloride because of its greater solubility and the consequent density of a saturated solution, which sinks to the bottom of the water to be tested. A 50 c.c. stoppered flask is filled almost full with the water to be tested, and another similar flask with some of the same water, which has been shaken with air until saturated with atmospheric oxygen. To each flask a pinch of chloro-quinol is added, and then 0·5 c.c. of a saturated solution of ammonium bromide containing 10 per cent. of ammonia. The flasks are stoppered and shaken, and the colour of their contents compared after ten minutes. The aerated water becomes brownish yellow at once, the precise colour at first being to some extent dependent on the temperature, but at the end of ten minutes it assumes a red-brown tint, the intensity of which is independent of the temperature. The water under examination is shown to be free from or saturated with dissolved oxygen if it remain colourless or develop colour equal to that of the aerated water. If it develop some colour, but less than the aerated sample, its content of oxygen may be approximately estimated by direct comparison, or more accurately by comparison with a salt solution of known strength, which is saturated with air and then tested as described. The solubility of atmospheric oxygen in normal, thrice normal, and saturated sodium chloride solution at various temperatures is well known, and such solutions serve as standards. G. C. J.

Titrimetric Estimation of Phosphorus in Small Amounts. L. T. Bowser. (*Amer. Chem. J.*, 1911, 45, 230-237.)—A number of methods for the estimation of small quantities of phosphorus have been proposed, the objection to most of them being that any silica which may be present reacts as phosphorus. The best method is that of Veitch, which the author has now developed and modified. The phosphoric acid solution may contain 0·1 – 0·01 mgrm. of P_2O_5 per c.c. A sufficient quantity of this solution is evaporated to a convenient bulk and treated with 0·5 grm. of ammonium nitrate. When the solution is ready for precipitation, it is important that its volume should be not less than 20 c.c., otherwise the precipitate may contain free molybdic acid. The molybdate reagent is next added, employing 1 c.c. of the reagent when less than 0·8 mgrm. of phosphoric anhydride is present, and 1·5 c.c. when a larger quantity has to be dealt with. Precipitation is effected at a temperature of 55° C., which must be maintained for an hour, and the precipitate is then allowed to stand for two hours. It is then filtered off on a small asbestos pad, about $\frac{1}{16}$ inch thick, and washed with the usual wash-solution of potassium nitrate The precipitate is titrated with a standard solution of potassium hydroxide not stronger than 0·02 *N*. The presence of notable proportions of silica in the phosphate solution has no influence on the results, but when very large quantities are present filtration becomes difficult. It is necessary, however, to correct the results by numbers obtained by conducting a blank experiment with the asbestos used. With quantities of phosphoric anhydride ranging from 0·5 to 1·0 mgrm., the average error does not exceed 3 per cent. J. F. B.

Testing Plaster of Paris. *Report of (German) Standards Committee.* (*Chem. Zeit.*, 1911, 35, 222-223.)—Proposals for uniform testing of plaster put forward by the Committee, acting in association with representatives from Switzerland, Hungary, and Russia. *Sifting.*—Four to five kilos of the sample are to be put through a sieve 60 × 35 × 12 cm., of 2 mm. mesh, 16 meshes per square centimetre. Soft lumps fall to pieces under this treatment and pass through, but coarse impurities remain behind. The sifted material only is to be used for the subsequent tests. *Gauge.*—A glass beaker, 6 cm. high by 7 cm. diameter, is tared, and charged with 100 grms. of water at 15° to 20° C. Plaster is then added by hand until it no longer sinks—that is, until the water-mirror disappears, and a thin, dry layer of gypsum remains visible for three to five seconds. The addition of plaster should be so conducted that this point is reached in from one and a half to two minutes. The beaker is again weighed, and the amount of plaster used is the measure of the gauge. *Slip.*—A quantity of plaster, equal to that used in the last experiment, is added during about thirty seconds to 100 c.c. of water, contained in a basin made by cutting a 12 cm. rubber ball in two. The mixture is well stirred, and then turned out on a glass plate, and given roughly the form of a cake, 10 to 12 cm. in diameter and

5 to 7 mm. thick. At intervals of half a minute a knife, with blade 2 mm. thick, is passed through the cake, cleaning it after each cut. When the walls of the channel thus carved cease to run together, the time (Giesszeit) which has elapsed since the first addition of plaster is noted. *Time of Setting.*—When the cake from the previous experiment shows, by its behaviour under pressure of a finger, that it is nearly set, slices 2 mm. thick are cut from it by means of a sharp knife at one-minute intervals. When the cut surface appears granular and crumbling, the time (Streichzeit) since first addition of plaster to water is noted. *Fineness.*—One hundred grms. are put through a 60×35 cm. sieve of thirty meshes per lineal cm., and 0·22 mm. actual mesh. Fine plaster leaves less than 10 per cent. of residue; medium, 10 to 12 per cent.; and coarse plaster, 20 to 30 per cent. *Tensile Strength.*—Four times as much plaster as was used in the earlier experiments is added to 400 c.c. of water at 15° to 20° C., and the resulting slip used to cast six figure-of-eight blocks, the cross-sectional area of which in the plane where they will be broken is 5 sq. cm. With quick-setting plasters it may be more convenient to mix half the quantities stated, and cast only three blocks at one time. After half an hour the blocks are removed from the moulds, and preserved in a dry room at 15° to 20° C. Blocks should be tested after one and twenty-eight days. *Bending Strength.*—This is also determined by a modification of the Michaelis apparatus, used for determination of tensile strength. Prisms $16 \times 4 \times 4$ cm. are used for the test. G. C. J.

Uniform Methods of Analysis for Portland Cement. F. Framm. (*Chem. Zeit.*, 1911, **35**, 257.)—A report presented to the general meeting of the Association of German Portland Cement Makers, as a result of experiments carried out in the Association's laboratory. The report includes directions as to sampling and the preservation of samples, as to the concentration of the necessary acids and other reagents used in the analysis, and as to the preparation and standardisation of solutions of permanganate, etc. After defining the means to be taken in the preparation of the small sample to be used for analysis, the report proceeds to give detailed directions for the estimation of loss on ignition, silica and insoluble matter, alumina and ferric oxide, lime, and magnesia. Methods follow for the separate estimation of matters remaining insoluble after successive treatment with dilute hydrochloric acid and 5 per cent. sodium carbonate, of sulphate, of iron, of sulphide sulphur, and of alkalis. Before these methods are promulgated by the Association as official, opportunity is to be given for testing and criticism.

G. C. J.

Separation of Strontium from Calcium. L. Moser and L. Machiedo. (*Chem. Zeit.*, 1911, **35**, 337-338.)—The authors have examined Rawson's method (*J. Soc. Chem. Ind.*, 1897, **16**, 113), which depends on the fact that strontium nitrate is but sparingly soluble in concentrated nitric acid. The solubility is, however, now shown to be as high as 20 mgrm. per 100 c.c. of nitric acid of 1·45 sp. gr., the most favourable concentration. Under the most favourable circumstances, therefore, 3 to 4 per cent. of the strontium will pass into the filtrate; and when the apparent error in actual separations falls below 2 per cent., as it may do, this is due to a partial compensation of errors; for, though pure calcium nitrate is readily soluble in nitric acid, in admixture with strontium nitrate the last traces of calcium nitrate stubbornly resist solution. Examination of other methods show that the ether-alcohol method of separation alone of existing methods has any claim to exactness. G. C. J.

Volumetric Estimation of Uranium. F. Ibbotson and S. G. Clark. (*Chem. News*, 1911, **103**, 146-147.)—The authors have used a simple form of the Jones "reductor" for the reduction of uranium solutions, after which they may be titrated with permanganate. The reductor consists of a 15 cm. column of 120 grms. of 20 to 30 mesh amalgamated zinc. With cold solutions, the authors found that five minutes' passage through this reductor was more than sufficient to insure conversion to the uranous condition, the solution being brownish-green instead of clear green, denoting a further stage. The reduction, however, could not be carried to the extent that only $U_2(SO_4)_3$ was present. If the brownish-green liquids, however, be treated by aspirating air through them for a few minutes, the pure green colour is obtained, and titration with permanganate gives correct results. Thus uranous solutions are stable in presence of air. The following method was therefore adopted, requiring only about fifteen minutes to carry out. The uranyl solution, containing from 2 to 5 per cent. by volume of free sulphuric acid, is heated in a flask with 50 grms. of pure, finely divided amalgamated zinc (20 to 30 mesh), till a dark brown colour is developed. The contents of the flask are rapidly cooled, and the cold solution poured through a small pulp filter, which is washed with cold water. Air is aspirated for a minute through the solution, and this is then titrated with $\frac{N}{20}$ permanganate. The zinc may be used a dozen times before reamalgamation is required. Since uranyl solutions are not reduced by sulphurous acid, a mixture of ferric sulphate and uranyl sulphate can be assayed by first reducing the iron with sulphurous acid, titrating with permanganate, and then treating the titrated solution as described.

A. R. T.

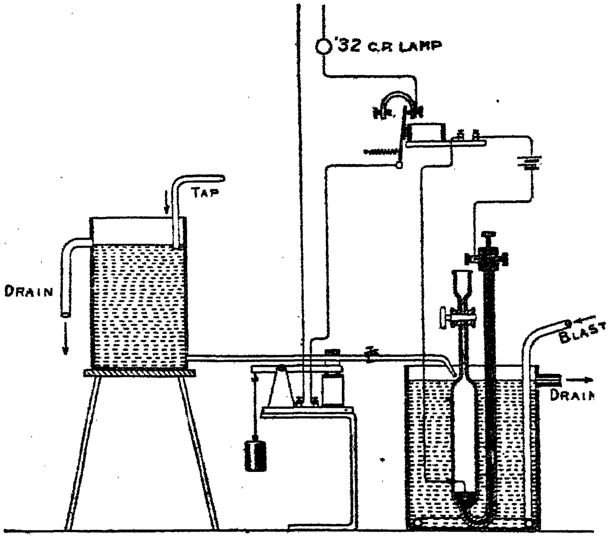
Estimation of Potassium Perchlorate in Potassium Chlorate. K. Scheringa. (*Pharm. Weekbl.*, 1911, **48**, 15-18.)—Sulphur dioxide reduces potassium chlorate to potassium chloride, but does not attack potassium perchlorate.

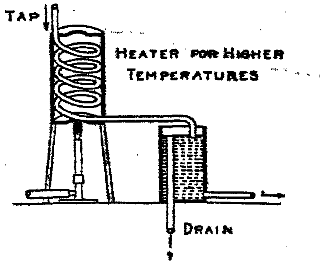
Use of Sulphur Dioxide in Checking Strengths of Volumetric Solutions of Iodine, Alkali, and Silver. E. Elvove. (*Amer. J. Pharm.*, 1911, **83**, 19-23.)—Pure silver is used as the standard, and solutions are prepared in the following order, each serving as a standard for the succeeding one: Ammonium thiocyanate (standardised against pure silver), silver nitrate, hydrochloric acid, sodium hydroxide, oxalic acid, potassium permanganate, sodium thiosulphate, and iodine. The strength of the iodine solution may be checked by reduction with sulphur dioxide and titration of the iodide after neutralisation.

Gravimetric Estimation of Tellurium and Alkalimetric Estimation of Telluric Acid. A. Rosenheim and W. Weinheber. (*Zeitsch. anorg. Chem.*, 1911, **69**, 266-269.)

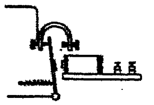
APPARATUS, ETC.

Constant-Temperature Bath for Use at Temperatures both Above and Below that of the Room. J. L. R. Morgan. (*J. Amer. Chem. Soc.*, 1911, **33**, 344-349.)—The bath is the vessel containing the large thermo-regulator, and the principle of the method is to deliver cold or hot water to the bath according as the desired temperature is below or above that of the room. The illustration shows the bath arranged for use at a temperature slightly below that of the room. The bulb of the regulator is 15 cm. by 3 cm., and the lower part of the other limb of the U is capillary (6 mm. external, 0·75 internal diameter). This capillary portion is only





HEATER FOR HIGHER TEMPERATURES



RELAY FOR HIGHER TEMPERATURES

6 mm. long, and the bore of the rest of the limb is 4 mm. Enough mercury is added to the regulator to reach half-way up the capillary, and the bulb is then filled with toluene as usual, the cock being left open. A platinum wire is fused through the glass of the regulator near the bottom, and a threaded brass collar is attached to the wider continuation of the capillary tube. Through this collar passes a threaded brass rod (2 mm. diameter), to the lower end of which is soldered a piece of platinum wire, 0·5 mm. in diameter, which makes contact with the mercury. These terminals of the regulator are shown, put in series with a dry cell and a relay. This relay is so

arranged that the house circuit, in series with a 16 c.p. lamp, can be either made or broken in a large telegraph sounder connected to the other two terminals of the relay. This sounder has attached to it a brass bar, about 5 mm. above the lowest position of the arm, when it is attracted by the magnet. The other end of the sounder arm has attached to it by a wire a weight of several pounds, so that when no current is passing through the sounder, the arm presses tightly against the brass bar. The thin rubber tubing of 4 mm. bore connecting the constant level supply of cooling water with the bath is run between the sounder arm and the bar above it. Cold water is allowed to flow from the constant level supply to the bath until the requisite temperature is attained. The cock above the toluene limb of the regulator is then closed, and the threaded brass rod screwed out until the platinum point no longer makes contact with the mercury. The weight falls, the tubing is pinched, and the water supply is cut off, and remains cut off until the temperature rises sufficiently to complete the circuit once more. For temperatures above 50° C., it is most convenient to surround the bath by a water-bath several degrees above 50° C., and to regulate by using tap-water as cooking-water, as already described. For temperatures between room temperature and 50° C. regulation is best effected by supplying a regulated supply of heated water; and for temperatures below 10° C. it is found most convenient to surround the bath with an ice pail and supply it with a regulated supply of tap water to warm it to the required temperature. In these last two cases the relay needs to be so arranged that the supply of warm water is cut off when the temperature exceeds that required—that is to say, the passage of a current through the relay must break, not make, the sounder circuit. The principle is applicable to comparatively small baths, which may be kept within a few hundredths of a degree of any temperature between 1° and 90° C. Stirring is most easily effected by blowing air, by means of a water-pump, through a coil of tin pipe bored with four small holes. G. C. J.

Still Water Gas Calorimeter. J. H. Coste and B. R. James. (*J. Soc. Chem. Ind.*, 1911, **30**, 258-261.)—Fig. 1 shows the gas-holder, Fig. 2 the calorimeter. The gas-bottle A contains $\frac{1}{10}$ cu. ft. from the point of the metal-rod G, to the bottom of C. The bottle being nearly full of gas, the burner (attached to E) is lighted; and when the level of the water approaches G, the short length of rubber tubing by M is pinched, so that water can only pass by the small by-pass. This makes it possible to determine with sufficient accuracy when the water-level reaches G. The burner is then swung under the calorimeter, and the pressure on the rubber tubing released. The calorimeter proper, B, is of copper, nickel-plated, and polished externally and supported on wooden blocks within the surrounding annular vessel A, from which it is separated by a $\frac{1}{2}$-inch air space. The outer vessel and the calorimeter are filled with water at the same temperature. A central cylindrical chimney, C, of corrugated copper tubing rises from the bottom of the calorimeter and opens into a flat, oval copper box, DD, from the ends of which depend two very much flattened corrugated copper tubes, EE. Suitable deflectors separate the products from the air passing in to support combustion. A long, narrow water-channel, F, separates the side or exit chimneys from the central one, which is the combustion chamber. Strips of copper, GG, divide the ascending current of air and the ribs of copper, HH, promote a rapid transfer of heat from the place where the heated gases first strike the metal to the cooling water. The calorimeter holds 2·1 litres of water, the exact amount of which is fixed by the coincidence of its surface with the silvered point I, depending from the teak-bridge J, which rides on the edges of the vessel, and carries on one side the thermometer and on the other the bearing for the stirrer. KK represents a double horseshoe stirrer, so designed as to divide the water into many small currents. Gas is delivered to the burner M at a uniform rate (about $\frac{3}{4}$ cubic foot per hour for ordinary illuminating gas) and a thermometer divided on an open scale to $\frac{1}{10}$° C. is used to estimate temperatures to the nearest $\frac{1}{100}$° C.

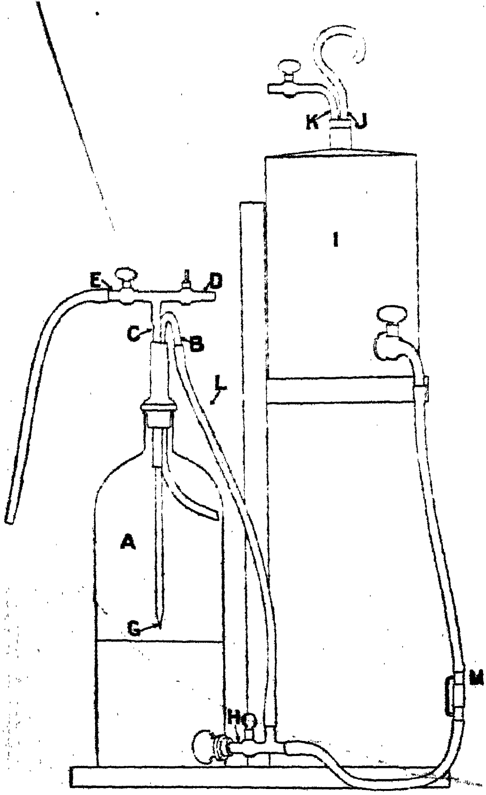


FIG. 1.

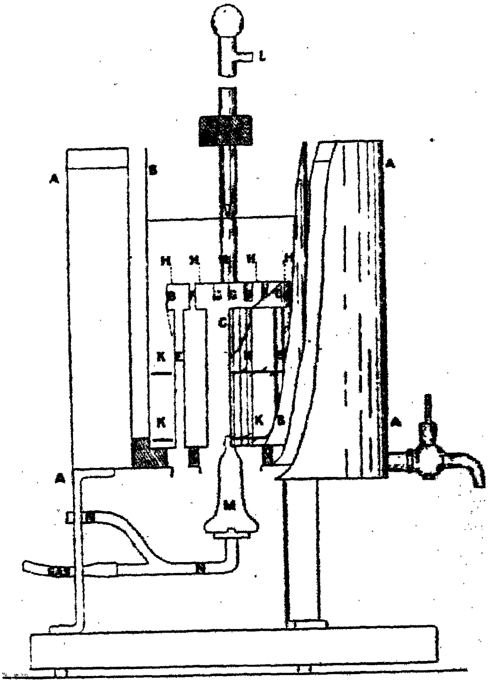


FIG. 2.

G. C. J.

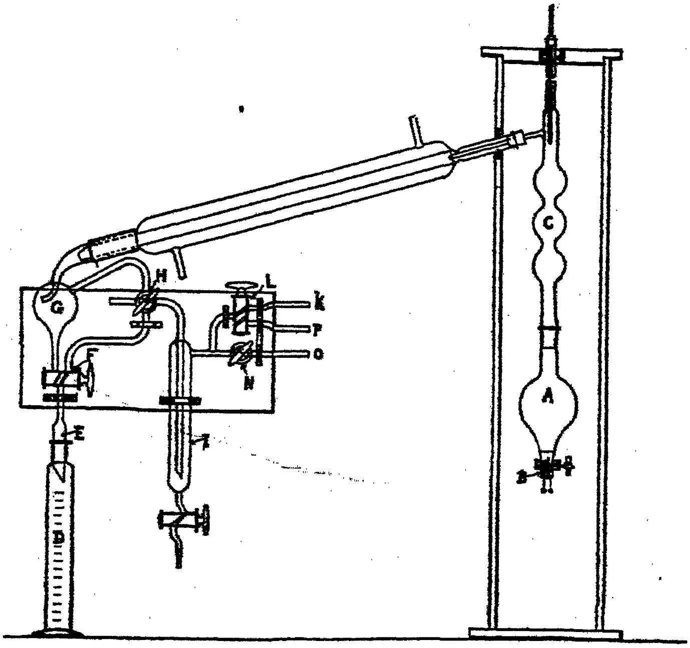
Capillary Analysis. J. Traube. (*Ber.*, 1911, **44**, 556-560.)—The investigation of capillarity constants is one of the simplest and most important of the physical methods applicable to analytical chemistry. The influence of various dissolved bodies on the surface tension of the solvent may frequently be employed for estimations—*e.g.*, of fusel oil, the concentration of alcohol, acetic acid, etc., also for purposes of diagnosing diseases. The measurement of surface tension by means of the capillarimeter and stalagmometer may be utilised with most satisfactory results for the determination of distribution and adsorption coefficients. Many colloidal media —*e.g.*, solutions of dyestuffs such as night blue, Nile blue, wool violet, also lecithin and soap emulsions, suffer a change in their surface tensions (size of droplets) when substances are added which can be described as "colloid poisons." These colloid poisons are identical with blood poisons, and include anions such as iodine, thiocyanate, perchlorate, etc., and cations, as mercury, lead, copper, and many other metals; bases such as alkaloids, snake poisons, acid bodies, phenol, arsenious acid, and many others. This method, using night blue and wool violet, has been employed for the technical estimation of nicotine. Minute quantities of iodine may thus be estimated in presence of large proportions of chlorine, bromine, and other ions—*e.g.*, in mineral waters. The iodine ion behaves quite differently to iodine in organic compounds, which fact is of value in physiological analysis. The method may be applied in the titration of poisonous ions in very dilute condition. If a solution of night blue be mixed with a few drops of mercuric chloride, the solution may be titrated with potassium iodide, and the capillarity constant (drop numbers) of the dyestuff solution will tend to be restored to the original value when the titration is complete. This principle is of very wide application. Alkaloids may be similarly titrated in a medium of wool violet, using tannic acid for the titration.

J. F. B.

Multiple Carbon Dioxide Recorder. (*Chem. Engineer*, 1911, **13**, 62-64.)—This recorder is designed to make continuous determinations of the carbon dioxide content in furnace gases, and in its several sizes will make separate and simultaneous records from one up to ten boilers. A continuous stream of gas is drawn through each section of the multiple machine by a single motor-driven suction fan, and a motor-driven rotary pump handles the paraffin oil in the successive operations which constitute a cycle for measuring off a sample of gas, the absorption of the carbon dioxide contained in it, the recording of this amount, and the expulsion of the sample. A single clock mechanism operates the whole series of drums, one to each boiler, which carry the record charts. The over-all dimensions of a four-record machine are 42″ × 13″ × 39″ high. The only adjustment necessary is setting the pen to zero when running on air. The caustic soda solution needs renewal once in ten days, when the machine is kept running night and day, each section making twenty records an hour. The paraffin oil is never changed, but a filter forms parts of its cycle. The pens are self-inking, and the ink-wells need refilling once a week. The apparatus is so designed that temperature errors are eliminated. The makers are the Sarco-Fuel Saving and Engineering Co., New York. G. C. J.

Extinguishing the Flame of Petroleum Spirit. F. Rathgen. (*Chem. Zeit.*, 1911, **35**, 308.)—The flame of 3 c.c. of petroleum spirit ignited in a basin may be extinguished by means of a simple device constructed upon the principle of the apparatus of the Fabrik Explosionssicherer Gefässe, Salzkotten. A solution of 15 c.c. of sodium carbonate in 200 c.c. of water, to which has been added 1 to 2 c.c. of liquorice extract, is introduced into a wide-mouthed half-litre flask, the cork of which is wired down. In the cork are two openings, through one of which passes a tube about 1 cm. in diameter, the inner portion of which extends a little way into the flask, whilst the other limb outside is bent downwards at an acute angle. Through the other hole passes a glass tube, also about 1 cm. in diameter, the end of which is a thin bulb. This tube is charged with about 15 to 20 c.c. of hydrochloric acid, and its other end is sealed up. On pushing this tube sharply downwards, the bulb will be shattered against the bottom of the bottle, and a stream of froth will issue from the other tube, and, when directed upon the burning petroleum spirit, will extinguish the flame. C. A. M.

Electrically-Heated Vacuum Fractionation Apparatus. H. S. Bailey. (*J. Amer. Chem. Soc.*, 1911, **33**, 447-450.)—The vessel A, in which the liquid is boiled, is of about 150 c.c. capacity, when 50 c.c. quantities are to be distilled, and its shape so chosen that distillation may be continued until only 5 c.c. remains, the line above the coil being a 5 c.c. mark. The heating coil B is of German silver wire, attached to stout platinum leads, which are fused through the glass. Connection between A and the fractionating head C is made by means of a ground joint. The connections K, O, and P connect to the house-pump, a Chapman and a Geryk pump respectively. The Geryk pump alone would suffice; but the use of the other pumps to exhaust most of the air at the start saves time, and, as a rule, oil-immersion pumps work better when they have only a small quantity of air to handle. The whole apparatus is exhausted, finally, by means of the Geryk through P. When it is wished to change the receiver D, cock F is reversed (it is shown



reversed in the figure), thus disconnecting G and D, and connecting E with the lower limb of the cock H. H is then given a half turn, which disconnects the distillation system from the pump, and puts E in connection with the atmosphere D is replaced by another receiver, cock H so turned that this receiver is connected to the pump, and when it is exhausted F is again reversed, and the contents of G allowed to flow into it. I is a trap to catch any liquid, which, in case of accident, might get sucked through the pump connections. G. C. J.

GOVERNMENT REPORTS.

I. On the Bleaching of Flour and the Addition of So-called "Improvers" to Flour; by J. M. Hamill. II. On the Chemical Changes produced in Flour by Bleaching; by G. W. Monier-Williams.

(Reports to the Local Government Board: Food Report No. 12, 1911, 1-65.)

The practice of bleaching flour originated in this country about ten years ago, and was rapidly adopted by millers in the United Kingdom, in America, and elsewhere. Many patents have been granted for bleaching processes and for the application of various chemicals to the bleaching of flour. Ozone, chlorine, oxides of chlorine, bromine, nitrogen peroxide, etc., have been suggested and tried as bleaching agents, but of these substances nitrogen peroxide is the only one which has given satisfactory results, and all commercial bleaching of flour at the present time depends on the use of this substance. The nitrogen peroxide is prepared either chemically or electrically; the electrical method produces a mixture of nitrogen peroxide and ozone, of which the former gas is the active bleaching agent, as it is now generally believed that ozone has no bleaching effect on flour. Inquiries made in connection with the preparation of this Report elicited the fact that bleaching in this country has not yet become universal, even amongst the larger firms; many millers have never bleached flour for sale, and others have abandoned the practice. The precise effect upon flour of bleaching by nitrogen peroxide is a matter of considerable dispute amongst millers and bakers, but the most obvious effect is the change of colour produced; other more subtle changes, such as alterations in the baking qualities of the flour, are also attributed to its use. It seems probable, however, that bleaching, when properly carried out, produces no appreciable effect on the baking qualities of flour, and there is no sufficient evidence that mere bleaching can counteract defects in flour due to unsoundness in the wheat from which it was milled. As regards the detection of bleach in flour, the chemical change most easily demonstrated in flour as a result of bleaching is the formation of nitrites, and the Griess-Ilosvay reaction (ANALYST, 1906, 31, 300) affords an exceedingly delicate test for detecting the presence of nitrites. As flour may absorb traces of nitrous acid from the atmosphere, the detection of mere traces of nitrite in a sample is not sufficient evidence that the flour has been bleached with nitrogen peroxide; only when the quantity of nitrite exceeds 1 part per million does the inference of bleaching amount to practical certainty.

From the results of experiments, it appears that bleaching has a distinctly inhibitory effect on the peptic digestion of flour, but no observable effect on the pancreatic digestion; it is also pointed out in the Report that nitrites have a distinct action on the heart and vascular system, and that, consequently, it is undesirable that small doses of nitrites should be consumed in bread day by day for years.

The practice of adding "improvers," such as acid calcium phosphate, phosphoric acid, sulphur dioxide, phosphorus pentachloride, etc., is considered to be most dangerous, as the addition may be indiscriminate; the miller is not always aware of the nature and character of the "improver," and other persons besides the miller may also add a quantity of an "improver," so that by the time the flour reaches the consumer it may have departed considerably from its original state of purity.

The action of air containing nitrogen peroxide upon flour, in quantities up to 300 c.c. of nitrogen peroxide per kilo of flour, may be summarised as follows:

1. The golden-yellow tint of the flour is destroyed. After being kept a few days, the more highly bleached samples became decidedly yellow, whilst those treated with from 30 to 100 c.c. of nitrogen peroxide per kilo became still whiter, the maximum bleaching effect being attained within these limits.

2. From 30 to 40 per cent. of the quantity of nitrogen peroxide used is absorbed by the flour. After the lapse of several days, the proportion of nitrites present decreases considerably in the higher concentrations, but remains fairly constant in the slightly bleached samples.

3. Approximately 60 per cent. of the total nitrogen introduced as nitrogen peroxide into the flour may be recovered as ammonia a short time after bleaching by reducing the aqueous extract of the flour with a copper-zinc couple.

4. In highly bleached flours a considerable increase in the amounts of soluble proteins and carbohydrates takes place. If 1 kilo of flour is bleached with 300 c.c. of nitrogen peroxide, the amount of soluble protein is doubled. This appears to be due to the solubility of gliadin in nitric acid.

5. If highly bleached flour is allowed to stand for some time after bleaching, the oil undergoes very considerable alteration, and acquires the characteristics of an oxidised oil. About 6 per cent. of the nitrogen introduced is absorbed by the oil.

6. The absorption of nitrogen peroxide by flour does not appear to be accompanied by the production of free nitrogen or diazo-compounds.

7. Sodium nitrite is not found to exert any inhibitory effect on the digestion of soluble starch by saliva, but the rate of digestion is considerably retarded if the starch has been treated previously with nitrogen peroxide. Bleaching also retards the salivary digestion of flour.

The opinion is expressed that it is undesirable that such an indispensable foodstuff as flour, the purity and wholesomeness of which are of first importance to the community, should be manipulated and treated with foreign substances, the utility of which, from the point of view of the consumer, is more than questionable.

W. P. S.

II. On the Presence of Calcium Sulphate in Baking-Powder and Self-Raising Flour. Dr. J. M. Hamill. (*Reports to the Local Government Board, Food Report* No. 13, 1911, 1-16).—The Report deals with the following points: 1. The nature and varieties of baking-powder. 2. The possible sources of calcium sulphate in baking-powders. 3. The strength and preparation of acid calcium phosphate. 4. Formulæ for compounding "phosphate" baking-powders. 5. Comparison of "phosphate" and tartaric baking-powders. 6. Self-raising flour. 7. Consideration of arguments in defence of the presence of calcium sulphate in acid calcium phosphate.

Samples of acid calcium phosphate were found to contain amounts varying from 1 to 56 per cent. of calcium sulphate, as an impurity, the latter quality being used to a considerable extent by makers of self-raising flour. Less commonly this low-grade phosphate is employed in baking-powders sold to the public. The consumer derives no benefit, and is liable to be prejudiced by the use of such material. The existence of an understanding as to the degree of impurity which would entail the rejection of such low-grade phosphates would serve alike the interests of the public and the majority of traders. Apart from any administrative action which may be recommended to public authorities on this matter, it is believed that the following recommendations would be welcomed by many traders:

(*a*) The lowest grade of acid phosphate made by manufacturers to be used as a food ingredient to carry a guarantee that it does not contain more than 10 per cent. of calcium sulphate. Phosphate preparations should not be sold under names suggesting that they contain tartaric acid or cream of tartar. (*b*) Bakers, self-raising-flour makers, and others using acid phosphates, should, if using the lower grades, in all cases stipulate that calcium sulphate is not to exceed 10 per cent. (*c*) Vendors of baking-powders containing acid phosphates should stipulate that the powders do not contain a higher proportion of calcium sulphate than would be contributed by a commercial acid phosphate containing 10 per cent. of calcium sulphate. They should not receive or sell such powders under descriptions which may lead the purchaser to believe that he is buying preparations of tartaric acid or cream of tartar.

Action by Local Authorities under Sale of Food and Drugs Acts.—It would seem desirable that local authorities under these Acts should invite reports from the public analyst as to proportions of calcium sulphate in a series of samples taken in the district of (*a*) baking-powders which appear to contain acid phosphate of lime; (*b*) acid phosphates supplied to confectioners, etc.; (*c*) self-raising flours. When necessary, the institution of proceedings under Section 6 of the Sale of Food and Drugs Act, 1875, should be considered. In an Addendum, C. H. Cribb reports on the alleged utility of calcium sulphate in "phosphate" baking-powders. He finds no justification for its presence. H. F. E. H.

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REVIEWS.

Allen's Commercial Organic Analysis. Vol. II.: Fixed Oils, Fat, and Waxes. Edited by Henry Leffmann and W. A. Davis. Fourth edition. London: J. and A. Churchill. 1910. Pp. x + 520. Price 21s. net.

The scheme adopted in the preparation of this volume doubtless presents great advantages, and the writing of articles by those particularly versed in the subjects discussed makes for accuracy and efficiency; nevertheless, one misses the easy style of Allen and there is no longer the same adherence to a general plan, which can only be attained when the whole work is the product of one brain.

Differences of nomenclature occur, such as the use of the term "linolic acid" by Mitchell and Archbutt in their respective sections, and "linoleic acid" by Klein and Robertson; instances of repetition are to be found—for instance, both Mitchell (p. 38) and Gill (p. 512) describe Mackey's "cloth-oil tester"—and there are omissions, the most serious of which is the absence of methods for the examination of candles (apart from wax candles) and candle material. Probably the difficulties of dual editorship in two countries has led to this result, though there is evidence in the numerous cross references that the editors have endeavoured to carry out their onerous task.

Allen was unique; a great scientist, a thoroughly sound chemist, he was, first and foremost, a consulting analyst, holding appointments under the Sale of Food and Drugs Acts, and being engaged as adviser in many industries. He wrote from the point of view of the analyst, and his book was one to which an analyst could turn to find just the information and advice he wanted. The present volume is written by many contributors, some well known and others whose names are not so familiar; but while all have acquitted themselves well, none of them write quite from the same point of view as Allen.

The first portion is by C. A. Mitchell on general properties and analytical methods, and gives a general review of the examination of oils and fats; while it is quite correct to term the radicle $CH_2.CH.CH_2$ *glyceryl*, there is a serious objection to calling it *glycyl*, as this term is now used for the radicle of glycine or amino-acetic acid. The usual methods for the examination of oils and fats are described in quite sufficient detail. It is to be regretted that Hanus' method for iodine absorption estimation is not mentioned, seeing that Hanus' figures are frequently quoted in literature. One or two of the methods mentioned, such as electrical conductivity and heat of combustion, might have been omitted, as no useful information is given about these.

The section on special characters and modes of examining fats, oils, and waxes, by L. Archbutt, is excellent, and perhaps more nearly approaches Allen's style than any other portion of the book. The method of estimating paraffin in waxes by treating the unsaponifiable matter with acetic anhydride is not, however, described; neither in this nor the preceding section is any mention made of chaulmoogra oil nor the interesting series of acids containing closed rings described by Power and colleagues.

Cecil Revis and E. R. Bolton contribute an article on butter-fat. The subject is treated with some originality, a selection only of the numerous methods being given; but the selection appears judicious. The writers attach special importance to the method of Avé Lallemant. The phytosteryl acetate test, an excellent modification of which is given, might have been described in a little more detail, the directions being meagre for those not used to the method. This section is perhaps the best in the book; these writers might with advantage have been entrusted with much more.

C. A. Klein writes on linseed oil, and gives a large amount of technical information.

Higher fatty acids are treated of by W. Robertson. This section might well have been amplified by the inclusion of a notice of the solid acids prepared by various methods from olein, and by some description of the products used in the candle industry.

The section on soap is by Henry Leffmann. This is purely analytical, and probably the technical side has been purposely left alone. The article is well written, and space is economised by the numerous cross-references. It may be modesty on the part of the writer which has led him to omit the useful rapid method for the valuation of soap, by measuring the fatty acids separated from a 10 per cent. solution in a Leffmann-Beam bottle.

The other co-editor, W. A. Davis, contributes an article on glycerol. The subject is treated of in an efficient manner; but it might have been mentioned that Hehner's method is by far the simplest for the estimation of glycerol in oils and fats.

Cholesterol and phytosterol are treated of by J. A. Gardiner, but hardly in a manner which is of use to the analyst; it would have completed the study if some mention had been made of coprosterol. Some of the melting-points in this section are evidently misprints.

The volume is concluded by a section on wool-fat, suint, and dégras, by A. H. Gill, and the article on cholesterol might well have been combined with this. The writer has treated this difficult subject in a lucid manner.

H. DROOP RICHMOND.

THE CHEMISTRY AND TESTING OF CEMENT. By CECIL H. DESCH, D.Sc., Ph.D.
Pp. x+267. London: Edward Arnold. 1911. Price 10s. 6d. net.

In view of the progress which our knowledge of the chemistry of cement has made in recent years, the present volume by Dr. Desch is very welcome, presenting as it does quite a clear and readable account of a subject which, to the large majority at least, has hitherto been somewhat obscure. The work under review "explains the nature of the chemical compounds contained in cements, the reactions involved in their preparation from the raw materials, the changes which take place during setting and hardening, and the methods of controlling, both chemically and mechanically, the quality of the product." After a brief historical introduction and a short but sufficient discussion of raw materials and manufacturing processes, the author confines himself mainly to the most recent and best views of the constitution and properties of the various calcareous cements, ignoring, as we think rightly, the old-fashioned and crude theories. Methods of thermal analysis and microscopical examination are shown to have been mainly instrumental in developing our knowledge of the constitution of cements, while the setting and hardening processes are now recognised as occurring in a colloidal medium. The physical and mechanical properties of cement and cement products are ably discussed in the light of recent knowledge.

The analytical section is perhaps somewhat brief, but the methods given are, as a rule, straightforward and reliable, and described with sufficient fulness for any competent chemist to carry out. Here, as indeed throughout the work, the author has used admirable discretion, and, unlike many others, has withstood the temptation to include fancy alternative methods of doubtful utility. The Schrötter method of determining CO_2 is, however, hardly worthy of a place in such a book. In the determination of manganese in slag, it will be found advantageous to remove alumina by means of caustic soda before separating iron as basic acetate. Moreover, few chemists will agree with the author in his preference for the sulphuric acid method of attacking insoluble silicates over the ordinary, but satisfactory, fusion method.

A description of the sliding weight machine for determining tensile strength would have been preferable to the older shot machine, as being more representative of modern practice. A misleading statement occurs on p. 143, where coarsely ground cement is said to weigh *less* (instead of *more*) than an equal volume of finely ground cement. The statement is contradicted both by the figures given by way of illustration and by a statement on p. 145. The formula for hydraulic calcium ferrite given on p. 72 as $3CaO.Fe_2O_3$ is apparently a misprint for $3CaO.2Fe_2O_3$.

Numerous references to recent investigations are given, and a discussion of the Revised British Standard Specification is appended. The book on the whole is excellent, is well published, and should be read with profit by everyone interested in the subject of cement.

GILBERT J. ALDERTON.

QUALITATIVE CHEMICAL ANALYSIS FROM THE STANDPOINT OF SOLUBILITIES, IONISATION, AND MASS ACTION. By J. I. D. HINDS, Ph.D., LL.D., Professor of Chemistry, University of Nashville and Peabody College for Teachers, Nashville, Tennessee. Pp. vii + 265. London: Williams and Norgate. 1911. Price $2.

A brief account of the physical chemistry necessary for the proper understanding of qualitative chemical analysis is given in the first four chapters of the book. Then follow the usual separation tables and tests for elements, acids, and bases. In the majority of cases the equations given to represent chemical changes are written in the ionic form, and this departure from the usual practice is certainly a good one, as it enables the student to realise what element or radicle of a reagent is concerned in the test for which that reagent is applied. Another good feature of this book is that the solubilities of many precipitates are given, and also the degree of ionisation of many acids in normal and decinormal solutions.

The phraseology is at times somewhat ambiguous, and shows a want of care in the preparation of the book which detracts from its value. Thus, on p. 39 we find the following: "Osmotic pressure may be regarded as the measure of the tendency of a solid to go into solution, and may be called *solution pressure*. The solution is saturated when the solution pressure and the osmotic pressure are equal." Again, on p. 183 we have the statement that "Phosphorus forms two regular oxids." Why regular?

Errors and omissions are rather numerous, and amongst the former we note on p. 215 the statement that calcium tartrate is insoluble in acetic acid, and on p. 211 that lead, aluminium, iron, and copper, form sparingly soluble acetates. With regard to the omissions, we find no mention of Denigès' test for citric acid, or of the resorcinol test for tartaric acid.

H. R. LE SUEUR.

BACTERIOLOGICAL AND ENZYME CHEMISTRY. By GILBERT J. FOWLER, D.Sc., F.I.C., Lecturer in Bacteriological Chemistry, Public Health Department, Victoria University of Manchester. First edition. Price 7s. 6d. net. Published by Edward Arnold.

This book, as is clearly stated in the preface, is an attempt to give a general outline of the chemical aspects of bacteriological and enzyme action. Such an attempt is undoubtedly beset with difficulties, as the various subjects are themselves of considerable extent, and it is only quite recently that our knowledge has become sufficiently exact to make such an attempt. The author has certainly succeeded in collecting together the greater part of our reliable knowledge on these subjects, which is scattered through much scientific literature, and the information is on the whole remarkably accurate and trustworthy; and it may be only an attempt to make the subject readable that is the cause of some lapses from strict scientific accuracy. This is particularly noticeable in the section dealing with the chemistry of the cell, where, for instance, osmotic pressure is said to be proportional in dilute solutions to the number of *molecules* present in the given volume; and exception might be taken on certain grounds to the definition of fermentation given at the end of the section. The chapters on purely chemical subjects are well and clearly written; and if, perhaps, a little unnecessarily full for the purpose of the book, they give all the information necessary to understand the subject of enzyme action, though a concise statement of the law of mass action as bearing on catalysis in general, and the reasons for the departure of enzyme action from this law, and also as foreshadowing reversibility, would seem to be a useful addition. It is a little unfortunate that, in the section on the action of amylase on starch, general reactions of proteins are set down as general reactions for enzymes, as the impression is given that the two are quite similar—a statement which, although quite intelligible to the expert mind, might give a wrong impression to the student. There is rather a tendency in the latter chapters to compression, and as these chapters are the most important from a student's point of view, the freedom of treatment noticeable in the earlier chapters would have been welcome. For instance, in the section dealing with the nitrogen cycle, though all the reliable data are given, there is lacking that correlation of the various bacterial agencies which modern research justifies, and which makes this subject so interesting. The same is true of the chapter on sewage purification, which, excellent in itself, lacks that dogmatic treatment which one expects from such an author, and which helps the student from being overwhelmed with conflicting views. Though in such a work the author must be free to choose his subjects, it is a little surprising to see the enormous amount of work done on nitrogen fixation in soils by bacteria, apart from "nodule" fixation, completely ignored, especially when "cyanamide" is referred to. The only really weak section is that on bacteriological technique; and it would have been better to have referred the reader to works on this subject or to have amplified the section considerably. The work is singularly free from spelling mistakes, though some, such as "absorption" for "adsorption" on p. 1, are a little unfortunate. The constitutional formulæ in the chapter on the chemistry of the albumins would be clearer if the various groups were separated in the usual manner. Concise and exact methods for testing for various enzymes, etc., placed at the ends of the various chapters, instead of the rather vague details scattered through the text, would probably render the work of greater use to the analytical chemist.

C. REVIS.

MODERN METHODS OF WATER PURIFICATION. By JOHN DON, F.I.C., A.M.I.Mech.E., and JOHN CHISHOLM, A.M.I.Mech.E. London: Edward Arnold. Pp. 368. Ninety-six illustrations. Price 15s. net.

The above volume may be confidently recommended to the attention of engineers and chemists interested in the question of water-supply, as a careful perusal shows it to be based on practical experience and a very thorough general study of the question.

Its value to the chemist will be found chiefly in the comprehensive outlook it affords on the practical side of the problem, and in the excellent summaries which are given of the present position of sundry important questions.

The subject of sand-filtration is treated with great care—in fact, it is possible that a discussion of the function of the filtering skin is somewhat over-elaborated; it, however, brings into relief the importance of the work of Director Pennink and his colleagues on the mechanical and biological phenomena of the sand-filter. It might have been of interest if a short account had been given of the actual experiments on which these views are founded, and which are described in a paper in *De Ingenieur* for July, 1905. An understanding of the true mechanism of sand-filtration is increasingly important in view of such developments as the non-submerged filters, operated by sprays in the same manner as sewage trickling filters. The work of Miquel and of Baudet on these lines is well described. With the exception of the experiments at Bedford some years ago, but little has been done in this country with filters of this type.

The chapters on mechanical filters and on water-softening processes are very complete, although some of the diagrams and descriptions are likely to be more readily understood by the mechanical engineer than by the general reader, interested chiefly in understanding quickly "how it works."

The important question of the use of ozone for water purification is very well handled, and is evidently the result of careful study. The advantage of the ozone treatment is, of course, that no added chemical is left in the purified water, and unless in the meantime considerable progress is made with the theoretically perfect process of sterilisation by ultra-violet rays, it is likely that more will be heard of the ozone method in this country.

The now classical researches of Houston on the effect of storage in eliminating the organisms of typhoid and cholera are very well summarised, as well as the same investigator's older studies on the important question of the action of different waters upon lead. In this section, again, it is evident that the authors have obtained first-hand information on the subject.

Methods of chemical and bacteriological examination of water are refered to, but in a somewhat sketchy manner, and the authors have evidently not set out to write for analytical chemists. Although practical methods for making rapid routine bacteriological examinations at waterworks are well described, it is singular that little or no reference is made to the importance of careful sampling, apart from which the results of chemical, and especially of bacteriological examination, may be of very little value. A useful account is given of the recently introduced method of testing a water-supply by determination of its electrical conductivity.

Of special interest, however, is the section on the microscopic examination of the "Plankton" life of waterworks, and it may be hoped that this will stimulate further inquiry in a very interesting field of research.

The authors are to be congratulated on having produced a living book on a subject of great importance to the wellbeing of modern communities.

G. J. Fowler.

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INSTITUTE OF CHEMISTRY.

Pass List: March—April Examinations, 1911.

Of thirteen candidates who presented themselves for the Intermediate Examination, nine passed: L. E. Campbell, B.Sc. (Lond.), R. B. Croad, A. J. Hale, B.Sc. (Lond.), W. F. Hollely, W. G. Prescott, C. J. Regan, B.Sc. (Lond.), C. B. Roos, B.Sc. (Lond.), H. B. Stevens, and A. F. Sugget. Of nineteen candidates who presented themselves for the Final Examination, twelve passed. In the Branch of Mineral Chemistry: W. O. Andrews, A.R.C.S. (Lond.), C. D. V. Georgi, B.Sc. (Lond.), H. L. Lucking, Ph.D. (Heidelberg), and O. D. Roberts; in the Branch of Metallurgical Chemistry: Gordon Wells, B.Sc. (Lond.), A.R.S.M., A.R.C.S.; in the Branch of Organic Chemistry: E. de Barry Barnett, B.Sc. (Lond.), G. E. Pool, B.Sc. (Lond.), and G. E. Slim; in the Branch of the Chemistry of Food and Drugs and of Water: S. W. Bridge, A. A. Eldridge, B.Sc. (Lond.), C. H. Hampshire, B.Sc. (Lond.), and H. M. Mason.

JUNE, 1911. Vol. XXXVI., No. 423.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE Ordinary Meeting of the Society was held on Wednesday evening, May 3, in the Chemical Society's rooms, Burlington House. The President, Mr. E. W. Voelcker, A.R.S.M., F.I.C., occupied the chair.

The minutes of the previous Ordinary Meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. A. D. Heywood, A.I.C., was read for the second time; and a certificate in favour of Mr. Horace C. Huish, 26, Westbourne Park Villas, Bayswater, W., chief assistant analyst to the Aylesbury Dairy Co., Ltd., was read for the first time.

Messrs. H. G. Battye, G. D. Elsdon, B.Sc., N. Evers, B.Sc., A.I.C., and A. W. Knapp, B.Sc., F.I.C., were elected members of the Society.

The following papers were read: "The Evaluation of Certain Spices used in Veterinary Medicine," by John C. Umney and Charles T. Bennett, B.Sc., F.I.C.; "The Detection of Traces of Hydrogen Cyanide," by G. Druce Lander, D.Sc., F.I.C., and A. E. Walden; "Note on the Composition of 'Bland's Pills,'" and "Note on the 'Pearl Coating' of Pills," by Albert E. Parkes, F.I.C., and John D. Roberts; and "A Reaction for Caulophyllin," by J. F. H. Gilbard, F.I.C.

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NOTE ON ALMOND AND APRICOT KERNEL OILS.

BY RAYMOND ROSS, F.I.C., AND JOSEPH RACE, F.I.C.

(*Read at the Meeting, April* 5, 1911.)

IN a paper by Lewkowitsch (ANALYST, 1904, 29, 105), the characteristics of almond and allied oils are discussed, but the fatty acids were not fully examined.

By the kindness of Messrs. Stafford Allen and Sons we were placed in possession of a sample of almond oil and one of apricot kernel oil of undoubted authenticity, and the following are the analytical results obtained:

	Almond Oil.	Apricot Kernel Oil.
Iodine value	98·3	101·4
Saponification value	194·7	192·4
Zeiss butyro-refractometer reading at 40° C.	57·5	57·5
Refractive index at 20° C.:		
Nd	1·4717	1·4717
Nc	1·4691	1·4690
Nf	1·4782	1·4783
Ng	1·4837	1·4838
Reichert-Meissl value	2·6	2·6
Polenske number	0·6	0·8
Acetyl value (true)	9·67	12·28
Specific gravity 15·5/15·5° C.	0·9197	0·9198
Shrewsbury and Knapp figure	18·7	22·0

INSOLUBLE FATTY ACIDS.

	Almond Oil.	Apricot Kernel Oil.
Iodine value	99·2	103·4
Saponification value	201·6	199·8
Zeiss butyro-refractometer reading at 25° C.	58·0	59·0
Refractive index at 25° C.:		
Nd	1·4644	1·4645
Nc	1·4622	1·4622
Nf	1·4710	1·4711
Ng	1·4761	1·4762

DISTILLATION OF FATTY ACIDS AT 40 M.M. PRESSURE—ALMOND OIL.

Fraction.	Zeiss (25° C.).	Iodine Value.	Saponification Value.
1	44·0	82·9	205·1
2	45·0	84·7	—
3	47·0	88·0	—
4	48·5	89·9	—
5	51·0	91·2	—
6	55·5	96·7	—
Residue	82·0	119·3	194·6

APRICOT KERNEL OIL.

Fraction.	Zeiss (25° C.).	Iodine Value.	Saponification Value.
1	46·0	85·2	203·7
2	47·0	87·1	—
3	49·0	89·6	—
4	49·5	90·3	—
5	52·0	93·1	—
6	57·0	97·5	—
Residue	86·5	129·6	193·7

The Bieber reaction was given by the apricot kernel oil, and even after being subjected to steam distillation for some hours, the residue gave the reaction strongly but not the distillate.

This colour reaction is therefore not due to a volatile constituent. Further, after keeping for a year and then bubbling air through the warm oil for three days the reaction was still strong, and as little as 5 per cent. could be detected when mixed with almond oil.

This test, according to our experience, is the only reliable one. The results given above certainly seem to confirm the opinion expressed by some authorities—viz., that the two oils are so similar in composition that for practical purposes they may be considered as identical.

We are informed from an authentic source that peach kernel oils, as such, hardly ever come on the market. Apricot and peach kernels are, however, often used together, and the oil thus obtained cannot be distinguished from apricot kernel oil.

The following table gives the limits of the figures obtained from the analysis of four samples of almond and three samples of apricot kernel oil bought commercially, and also the figures for one sample sold as peach kernel oil:

	Limits of Four Samples of Almond Oil.	Limits of Three Samples of Apricot Kernel Oil.	Peach Kernel Oil.
Iodine value	97 to 102	100 to 106	101·6
Saponification value	183·3 to 207·6	184 to 192·4	191·7
Specific gravity, 15° C./15° C.	0·9178 to 0·9199	0·9198 to 0·9200	0·9167
Zeiss butyro-refractometer reading at 40° C.	57·5° to 58°	57° to 58·5°	55·5°
Bieber reaction	nil	strong	strong
FATTY ACIDS.			
Saponification value	200·4 to 207	197 to 202	201·6
Zeiss butyro-refractometer reading at 25° C.	56° to 58°	57° to 59°	53°

Mr. C. A. Hill writes:

"In my experience the iodine value of apricot kernel oil is distinctly higher than that of almond oil. A number of samples of each during the past three years have given

	Almond Oil.		Apricot Kernel Oil.
Iodine value	93 to 98	...	100 to 110

"Although the analytical data afforded by almond and peach kernel oils are very similar, and may even overlap, I cannot concur with the opinion that on this account the oils may be considered as identical.

"The authors give Zeiss readings at 40° and at 25° C., and refractive indices at 25° and at 20° C. I think it far preferable to have one standard temperature for refractive indices of fixed oils, and that this should be 40° C."

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THE DETECTION OF TRACES OF HYDROGEN CYANIDE.

By G. DRUCE LANDER, D.Sc., F.I.C., and A. E. WALDEN.

(*Read at the Meeting, May* 3, 1911.)

SEVERAL important investigations relating to the detection and estimation of minute quantities of hydrogen cyanide have recently appeared. The colour reaction with alkali picrate has been most successfully employed by Guignard and by Armstrong in the study of enzymic action, whilst Waller (*Proc. Roy. Soc.*, B. 82, 1910, 574; ANALYST, 1910, 35, 406) has elaborated thereon a quantitative colorimetric process. The valuable work of Chapman (ANALYST, 1909, 34, 475, and 1910, 35, 469) elucidates the mechanism of the picrate reaction, showing it to be one of reduction, and therefore, being given by numerous other substances, to fail as a specific test for hydrogen cyanide. The papers of Berl and Delpy (ANALYST, 1910, 35, 362) and Lockemann (ANALYST, 1910, 25, 408) are concerned with the ferrocyanide test.

In view of the great sensitiveness of the picrate test, it appeared most desirable to make comparisons between it and the ferrocyanide reaction, having particularly in view the possibility of applying the latter specific reaction to such limiting cases as the detection of cyanide in the minimal quantities found in the brains of poisoned animals, as has been done with picrate by Waller.

Our experiments serve to show that, under regulated conditions, the ferrocyanide test is susceptible of equal, if not superior, refinement to the picrate, and that it may be applied with certainty and confidence to the above-named object.

As regards the limits of the various tests, Berl and Delpy have worked the ferrocyanide down to 0·00004 grm. hydrogen cyanide per c.c., and Chapman detects 0·00002 grm. with certainty by the picrate test. In Waller's numerous examples 0·00001 grm. appears to be the smallest amount actually estimated. Under defined conditions the ferrocyanide test recognises 0·00001 grm. with certainty, and definite positive results are obtainable with 0·000002 grm.

Berl and Delpy concentrate small quantities by extraction with ether, and subsequent resolution therefrom with a trace of alkali. This procedure is unnecessary, for we find that excessively dilute solutions can be boiled with alkali without change, as the following experiment shows: Ten c.c. of KCN solution required 18·8 c.c. $\frac{N}{100}$ I; after dilution to 150 c.c., addition of 6 drops of 10 per cent. caustic soda, and five minutes' boiling, the solution again required 18·8 c.c. of iodine. Since one almost invariably has to deal with distillates, this enables one to concentrate without risk of loss or the inconvenience of other dissolved solids.

In common with Berl and Delpy, we test in the cold, but we do not finally heat the alkali iron solution. No doubt many analysts have in their own experience noticed the advantage of working in the cold, but it is not the usual practice.

For quantities of less than 0·00002 grm. per c.c., we find the best method to be as follows: Add to the solution 2 drops of 10 per cent. caustic soda, and evaporate finally in a test-tube over the flame nearly to dryness; cool, add 1 drop of 2 per cent. ferrous sulphate, and leave in the cold for ten to fifteen minutes; add 2 to 3 drops of strong hydrochloric acid, warm gently, and cool. The liquid on careful dilution then shows a blue colour, the concentrated solution being generally blue-green. With a given concentration, better results are obtained thus than without evaporation.

With quantities of the order of 0·00002 grm. per c.c., boiling at once, after adding the alkali and iron, may give a negative result. Further, if a number of small distillates are separately taken and tested, or if a single large one is not evaporated, very appreciable quantities of cyanide may be entirely overlooked, and testing without evaporation will not give so good a result in any case as evaporation and subsequent dilution to the original volume.

The conditions governing the formation of sodium ferrocyanide from ferrous hydroxide and alkali cyanide are no doubt very complex. The reaction is not ionic, and the alterable character of the ferrous hydroxide further complicates the reaction.

The effect of concentration may be roughly gauged in its practical bearing from the following observations with 0·0025 per cent. hydrocyanic acid (as KCN), the tests being made without concentration:

Cyanide Solution.		Added Water.		Time.		Result.
(*a*) 1·0 c.c.	...	nil	...	10 minutes	...	good.
(*b*) 1·0 „	...	1·0 c.c.	..	10 „	...	positive.
(*c*) 1·0 „	...	2·0 „	...	10 „	...	blank.
(*d*) 0·5 „	...	0·5 „	...	10 „	...	blank.

The most remarkable factor is the apparent effect of the absolute quantity on the result of the test. Experiments (*b*) and (*d*) ought to have given the same result, the concentrations of cyanide being the same; and it may be mentioned that, as regards alkali and iron, these, in both instances, are in relatively enormous excess, the ferrous hydroxide further being a solid phase. We find similar effects with the picrate test. Quantities of 2 c.c., 1 c.c., and 0·5 c.c., of cyanide solution were mixed with equal volumes of Waller's reagent, sealed in tubes, and heated simultaneously at 37° C. for forty-eight hours. The 0·5 c.c. was distinctly less coloured than the 1 c.c. and 2 c.c. preparations. Considerations such as these serve to determine an absolute limit to microchemical tests.

The pale-coloured, weak alkali picrate of Waller will probably be preferred to the darker alkali hydroxide solution of Chapman, the colour of the latter tending to mask the dark yellow cyanide coloration. Thus, 0·000006 grm. KCN gave a definite result with 2 c.c. of Waller's reagent, but none with 2 c.c. of Chapman's reagent, whilst 0·000002 grm. gave a faint colour with 1 c.c. of Waller's reagent. The picrate test thus offers no advantage over the ferrocyanide in point of sensitiveness. Even if it did, there is less room for personal equation in the discrimination of pale yellow from blue-green, than of pale yellow from darker yellow.

Berl and Delpy have demonstrated that the ferrocyanide reaction affords excellent quantitative results. We do not place any reliance on the preparation of standard tints, being convinced that the excessive variations in quantity and concentration entirely vitiate results obtained by comparing an unknown quantity with an empirical standard. We recommend that, having obtained a certain result, it should be matched by taking known quantities of cyanide, evaporating, and treating in all respects exactly as was done in the original assay. In this way results, which in all such cases ought only to be regarded as approximations, are quickly obtained.

But such methods naturally need only be resorted to in limiting cases. For cyanide, we find that titration with $\frac{N}{500}$ iodine (freshly prepared) is useful; and since 1 c.c. of this equals 1 c.c. of $\frac{N}{1000}$ KCN (0·000025 grm.), it could be used for most of the cyanide determinations from blood and brain, such as are described below and by Waller.

That the ferrocyanide is equal to the picrate, if not superior, in toxicological work is shown by the following example: A dog, weighing 8·8 kilos, was destroyed by interthoracic injection of 3 c.c. of 8 per cent. KCN (equal to 0·1 grm. HCN). Ten c.c. of citrated blood, diluted with 30 c.c. of water, and acidified with tartaric acid, gave, on steam distillation, 15 c.c. of distillate, which yielded after concentration a ferrocyanide test amounting to the formation of a considerable precipitate, and equivalent approximately to 0·0001 grm.

An exactly corresponding picrate test gave excellent results. Ten grms. of brain similarly gave a good ferrocyanide result, equal to approximately 0·00002 grm., whilst the corresponding picrate test yielded results which were certainly positive, but not very well marked. The citrated blood was kept in a stoppered bottle for nine days, when 30 c.c., diluted with 50 c.c. of water, and acidified with tartaric acid, were slowly distilled in a current of steam, 10 c.c. being collected. Of this distillate, 5 c.c. required 6·8 $\frac{N}{500}$ iodine, corresponding to 0·00018 HCN, or 0·000036 HCN in 1 c.c. of distillate, or 3 c.c. of citrated blood. The ferrocyanide test was performed with 1 c.c. of distillate, and was matched by a comparison test from 1·7 c.c. of 0·002 per cent. HCN (as KCN), or by 0·000034 grm. HCN. This result is in sufficient agreement with the iodine estimation. In a second experiment, a dog was killed and its brain exposed eight days *post mortem*. Ten grms. treated as above gave an excellent result. The brain was left exposed to the air, and again tested after three and five days respectively. In both cases good positive tests were given, very much less than in the freshly opened specimen, but remarkably close to one another.

DISCUSSION.

Dr. WILLCOX said that he agreed with the authors that the Prussian blue test was more reliable than the picric acid test. In a recent instance in which he had compared the two methods, the picric acid test gave an excellent reaction, but unfortunately the colour soon faded; and it was afterwards found that the reaction was due, not to cyanides, but to sulphides, which would certainly be present in putrid viscera. Moreover, if it were necessary to prepare exhibits for production in court, the Prussian blue reaction was the more satisfactory one. When, however, the presence of hydrocyanic acid had been demonstrated, the picric acid method would be found useful for estimating the relative quantities present in different organs, provided they were not decomposed. He had found that the Prussian blue reaction was affected to some extent by an excess of ferrous sulphate, and he should like to hear whether the authors had found the addition of an excess of ferrous sulphate to be attended by any risk. It was, of course, necessary that the test should be carried out under constant conditions, and he felt sure that the authors were right in saying that, in making comparison tests with known amounts of cyanide, the complete process must be exactly followed, since the depth of the colour was considerably influenced by the conditions of working.

Mr. Chapman said that this paper was in a sense complementary to the paper which he had communicated to the Society recently, and in which he had called attention to the fact that the picric acid method was merely a very general reduction method, the red coloration being given by any substance capable of reducing picric acid in alkaline solution. From a diagnostic point of view the picric acid was on this account useless. The reaction was given not only by sulphuretted hydrogen, but by acetone, aldehyde, and a number of other substances which might quite conceivably be present in a toxicological distillate; so that, unless it were established that hydrogen cyanide was the only reducing agent present, no definite conclusion could be drawn from the results, and in toxicological work it would probably rarely, if ever, happen that one could be certain that no other reducing agent was present. The alkaline picrate method was undoubtedly useful in certain cases in which one required not so much absolute results as a series of comparative results. Under such circumstances the method had the advantage of being carried out even more quickly and easily than the Prussian blue method. The authors' results showed, however, that the Prussian blue test was actually more delicate than the picric acid method—a fact of which he (Mr. Chapman) was not aware at the time of the reading of his own paper. With regard to the method of titration with iodine, the same objection applied to this as to the picric acid method, since sulphuretted hydrogen and a number of other substances would react in the same way as hydrocyanic acid. He should like to ask the authors with what degree of certainty the Prussian blue method could be used—*i.e.*, whether one could always rely upon obtaining the same intensity of colour from a given quantity of hydrogen cyanide. With the picric acid method approximately the same amount of colour could always be obtained. He should also like to hear whether the Prussian blue standards were sufficiently stable. One advantage of the picric acid method was that the standards were easy to make, and could be kept for a reasonable time.

Dr. Lessing asked whether the authors were quite satisfied that no hydrocyanic acid was lost after boiling the liquid down to the required degree of concentration. It seemed possible that some of the cyanide might be converted into cyanate, and if this were so it might be the cause of the discrepant results obtained from the different absolute quantities in a bulk of 1 c.c. and 0·5 c.c. respectively. It would be interesting to know by what method the authors determined the absolute amounts of cyanide before starting and after concentrating, and suggested that it might be advantageous to use equivalent solutions of ferrocyanide as a standard, which would allow a conclusion as to the absolute quantities of cyanogen present.

Dr. Lander, in reply, said that in almost any case the proportion of precipitated iron hydroxide to cyanide in solution was very large. This was one of the complicated features of the reaction, and the physical mechanism of the production of ferrocyanide in solutions was not understood. Ferrous hydroxide was known to be slightly soluble, but, on the whole, the iron hydroxide in this reaction must be looked upon as a solid phase. Apart, however, from the slight coloration which it might impart to the final liquid, he did not think that the quantity of iron used was to be regarded as of very serious importance. He clearly recognised that the objections to the picrate process would apply equally to the iodine process. In both cases it was, of course, equally necessary first to ascertain that no reducing agent except hydrogen cyanide was present. He had suspected that cyanate might possibly be formed, but in experiments made by briskly boiling very dilute solutions no serious loss of cyanide was shown. Even supposing, however, that any formation of cyanate did occur—*e.g.*, by hydrolysis—during the evaporation, the results would still be comparable if the conditions of working were always the same.

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A REACTION FOR CAULOPHYLLIN.

By J. F. H. GILBARD, F.I.C.

(*Read at the Meeting, May* 3, 1911.)

Caulophyllin is a brownish-grey powder prepared from the Blue Cohosh (*Caulophyllum thalictroides*), the rhizome and roots of which were formerly included in the United States Pharmacopœia. It is prepared by precipitating the resins from an alcoholic tincture with water, and collecting and drying the precipitate. It is used chiefly by homeopathists and herbalists. According to Squire's "Companion to the British Pharmacopœia" (1908 edition, p. 349), caulophyllin has been recommended as an emmenagogue, sedative, diuretic and anthelmintic, and is best given in the form of a pill in doses of from 1 to 4 grains. It is also reputed to be used as an abortifacient (see Taylor's "Principles and Practice of Medical Jurisprudence," 5th edition, vol. ii., p. 167). A sample of caulophyllin which I examined contained:

Moisture (loss at 100° C.)	8·0
Matters soluble in 80 per cent. alcohol	72·5
Matters insoluble in 80 per cent. alcohol ...	19·5
	100·0
Total matter soluble in cold water	51·5

In the course of an investigation, in which I assisted Dr. Dyer, into the composition of certain pills which were stated to contain this drug, it became important to find, if possible, some specific test for caulophyllin, no distinctive test for its recognition having, apparently, been hitherto recorded. After several unsuccessful attempts the following test was arrived at: Five c.c. of an alcoholic extract, obtained by digesting 0·1 grm. of caulophyllin in 25 c.c. of 80 per cent. alcohol and filtering, are evaporated to dryness on the water-bath in a flat porcelain capsule. The residue is taken up with

1 c.c. of water, and broken up with a flat-headed rod. Two c.c. of concentrated sulphuric acid are then slowly added, and the mixture well stirred. An intense purple-blue colour is produced within five minutes. (With some specimens of caulophyllin the colour is more inclined to reddish purple.) When testing a mixture of drugs (as, for example, a compound pill), 0·1 grm. of the pill is repeatedly extracted with 80 per cent. alcohol, filtered, and the filtrate evaporated in a porcelain capsule. The residue is broken up with 1 c.c. of water, and 2 c.c. of concentrated sulphuric acid are added. If caulophyllin be present, a distinct blue colour will be shown at the edges after a few minutes. The reaction is best seen by daylight, or by the light of burning magnesium.

The reaction appears to be characteristic of this resin, or of some constituent of it. The resins of many other drugs which were tested at the same time did not give the reaction.

DISCUSSION.

Dr. WILLCOX congratulated the author on the discovery of this reaction. The colour was very characteristic, and was produced quite definitely in the case of a mixture. He thought that the reaction might possibly be produced by other substances, and he did not imagine that the author would claim for it that it was characteristic of caulophyllin and of nothing else. In the case referred to, however—in which he (Dr. Willcox) had also been concerned—the test afforded valuable aid, for while the reaction was not given by the pills in question, it was given with certainty by pills made up according to the formula which was stated to have been followed, this formula containing caulophyllin with other drugs.

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SULPHUR DIOXIDE IN COMMERCIAL GELATINS.

By HENRY LEFFMANN AND CHARLES H. LaWALL.

THE enforcement of the Federal Food Act, and similar acts, in most of the States of America has caused attention to be directed to the use of sulphur dioxide in certain classes of articles which have been treated with sulphur dioxide for antiseptic or bleaching purposes. Amongst such materials is gelatin, and considerable attention has been given to the occurrence of sulphur dioxide in this substance, and to the methods of determining it. It is recognised that simple methods of titrating with iodine solution may be misleading.

We have recently examined a number of commercial gelatins by the following method. A known weight of the sample was dissolved in water and distilled with phosphoric acid in an atmosphere of carbon dioxide, the distillate being received in an excess of iodine; the resulting sulphuric acid was then precipitated and weighed as barium sulphate.

It is evident from our results that with care in the selection and treatment of stock the manufacturer can keep the content of sulphur dioxide so low as to be negligible. Thus, of eighty-six samples from one factory, only two contained more than 50 parts of SO_2 per million, and six of the remainder not over 15 parts per million. Eighteen contained but traces, and most of the remainder less than 10 parts. From another factory, three samples were tested, all containing less than 10 parts per million. From a third source two samples, inferior in appearance and somewhat offensive in odour, contained respectively 305 and 331 parts per million.

The most striking results were obtained in the test of two samples of gelatin imported especially for bacteriological use, being the brand recommended by American bacteriologists. These samples contained 265 and 835 parts per million respectively of sulphur dioxide. It is obvious that an agent having so powerful a restraining action on micro-organisms will be particularly objectionable in a culture medium. It will be necessary, we think, for bacteriologists to establish a strict standard for gelatin.

It has been stated that gelatins low in sulphur dioxide are apt to be contaminated with bacteria, but this, if true, will have but little significance for the bacteriologist, as all media are sterilised.

PHILADELPHIA, U.S.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Examination of Absinthe and its Imitations. F. Schaffer and E. Philippe. (*Mitt. Lebensmittelunters. u. Hyg.*, 1911, 1, 1-13; *Chem. Zentralbl.*, 1911, 1, 1160-1161.)—According to the authors, the estimation of the "cloudiness coefficient" by dilution with water is not of much account, since it depends on the alcoholic strength, and the quantity and nature of the essential oils. In the detection of the poisonous thujone by Legal's reaction, the authors state that the test can be made sharper by adding the nitroprusside and sodium hydroxide and shaking, then waiting for half a minute before adding the acetic acid. Absinthe itself will barely show Legal's reaction; it is necessary to test a fraction rich in thujone, which occurs in the case of highly alcoholic liqueurs in the last fractions poor in alcohol, and in the case of weakly alcoholic liqueurs in the first fractions. A high percentage of alcohol weakens the thujone reaction. With pure thujone, working with fractional distillation of 100 c.c. of thujone solution, this body could be detected at dilutions of 1 in 50,000. The harmless carvone may also give a red coloration by Legal's test, likewise marc-brandy and denatured spirits. It is doubtful whether a really conclusive test for thujone has been found, and even then the presence of thujone would not afford conclusive evidence that wormwood has been employed in the preparation of the liqueur, since that ketone occurs in other plants. An essential oil prepared from the "great wormwood" gave Legal's reaction more strongly than one prepared from the "little wormwood" (*cf.* ANALYST, 1911, 12). J. F. B.

Colour Reactions of Aromatic Aldehydes and their Applicability in the Analysis of Spirits. T. von Fellenberg. (*Mitt. Lebensmitteluntersuch. u. Hyg.*, 1911, **1**, 311-350; through *Chem. Zentralbl.*, 1911, **1**, 1254-1255.)—The author has further investigated the reaction which takes place between the higher alcohols and a mixture of sulphuric acid and salicylic aldehyde (ANALYST, 1910, **35**, 403), and finds that this colour reaction is given by all unsaturated substances and by compounds which are converted into unsaturated substances by heating with sulphuric acid, unsaturated acids, in which the carboxyl group is next to the double bond, being exceptions. The reaction is also given by aldehydes and phenols, in which the para position is free; these phenols form colours of the type of rosolic acid. Although all aromatic aldehydes give the reaction, salicylic aldehyde has the advantage that it does not give a coloration with methyl or ethyl alcohol. Of the substances other than the higher alcohols, present in brandy and rum, only the terpenes, aldehyde, and acetal, give the coloration when treated with salicylic aldehyde and sulphuric acid. These three substances may be destroyed by boiling the spirit with silver oxide.

W. P. S.

Simplified Method for the Estimation of Arsenic in Atoxyl and Arsacetin. E. Rupp and F. Lehmann. (*Apoth. Zeit.*, 1911, **26**, 203-204; *Chem. Zentralbl.*, 1911, **1**, 1082-1083.)—The authors propose a simplified modification of the method of the German Pharmacopœia: 0·2 grm. of the substance is heated in a 200 c.c. flask with 10 c.c. of concentrated sulphuric acid at 70° C.; then 1 grm. of crystalline potassium permanganate is added in small portions, whilst the contents of the flask are rotated; 5 to 10 c.c. of officinal hydrogen peroxide are next added, drop by drop, until the liquid is colourless. It is then diluted with 20 c.c. of water, boiled for ten minutes, and further diluted with 50 c.c. of water. After cooling, 2 grms. of potassium iodide are added, the solution is allowed to stand for an hour, and then titrated with $\frac{N}{10}$ thiosulphate without an indicator. The authors quote examples showing that the results are in concordance with those of the official method.

J. F. B.

Preparation and Constitution of Caramel and its Estimation in Sugar Juices and Syrups. F. Ehrlich. (*Seventh Inter. Cong. App. Chem.*, 1909, Section V., 92-93.)—The author has succeeded in preparing a chemically homogeneous product of caramelisation by heating sucrose *in vacuo* in the oil-bath at 200° C., extracting the product with boiling methyl alcohol until colourless extracts were obtained, dissolving the residue in water, filtering and evaporating *in vacuo*. The product, amounting to 20 per cent., was "saccharan," having the formula $C_{[illegible]}H_{[illegible]}O_{[illegible]}$. It is the most intensely coloured "caramel" which has hitherto been prepared. It is therefore recommended as a standard for the colorimetric estimation of caramel, by dissolving 0·1 grm. in 100 c.c. of water and making observations in a 20 mm. cell. In this way the colouring power of any sample may be expressed numerically in terms of saccharan. The saccharan itself may also be determined by precipitating the other colouring matters by basic lead acetate, since saccharan is not precipitated by that reagent. Saccharan is a blackish-brown substance, existing in the form of lustrous scales; it is tasteless and non-hygroscopic. This substance may also be used as a standard in the colorimetric valuation of colour-malts and similar products, to which its shade approximates more closely than that of the iodine solution frequently employed. J. F. B.

Composition of Ripe and Unripe Bananas. K. Yoshimura. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, 21, 406-411.)—In order to ascertain the changes taking place in the composition of bananas during the ripening of the latter, analyses were made of the unripe fruit and of the same at intervals after it had been stored in a warm room; the results obtained were as follows:

	Unripe Fruit.	After One Week.	After Two Weeks.	After Three Weeks.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Water	70·13	70·31	71·47	70·72
	Per 100 Parts of the Dry Substance.			
Tannin	1·75	1·71	1·71	1·67
Starch	68·16	47·51	24·40	14·48
Reducing sugars	—	5·55	13·47	19·83
Cane Sugar (calculated as invert sugar ...)	—	14·92	28·61	30·12

As the quantity of tannin remains practically constant during the ripening process, it is probable that this constituent is without influence on the process. The conversion of starch into sugar proceeds fairly rapidly; it is at first changed into sucrose, but the latter is subsequently inverted by the action of the invertase which is present, so that there is no definite relation between the quantities of invert sugar and cane sugar formed. Sugars other than cane and invert sugar are not present in ripe bananas. W. P. S.

Analysis of Coca Leaves. A. W. K. de Jong. (*Arch. Pharm.*, 1911, 249, 209-214.)—In reply to certain statements of Bierling, Pape, and Viehöver upon the analysis of coca leaves (ANALYST, 1910, 35, 397) the author asserts that the persistent emulsion formed when petroleum spirit is used as the solvent may be resolved by expelling the petroleum spirit by means of a current of air. Comparative estimations have shown that somewhat higher results are obtained by the use of petroleum spirit instead of ether (*e.g.*, 1·47 as against 1·42 per cent.). Methods in which the alkaloid is extracted by shaking the leaves with the solvent never give correct results, and it is essential to use either percolation or extraction in a Soxhlet apparatus. The statement that the amount of water added in De Jong's original method (ANALYST, 1909, 24, 99) prevents complete extraction of the cocaine by the ether, is shown to be incorrect, the addition of water actually causing a slight increase in the yield. Two extractions with ether are quite sufficient to extract the alkaloid, and a third extraction is unnecessary. Petroleum spirit removes also δ-isatropylcocaine, although only very slowly. In the author's experience titration, as advocated by Bierling and his co-workers, will not as a rule give accurate results, since the alkaloid is a mixture of bodies of different molecular weights. For instance, in the case of Java leaves the alkaloid consists, in the main, of cinnamylcocaine and isatropylcocaine, the factor for which is not 0·00303 but 0·00329. The results obtained by the gravimetric and volumetric methods will therefore differ. C. A. M.

Estimation of Husk in Cocoa Powder. W. L. Dubois and C. I. Lott. (*J. Ind. and Chem.*, 1911, 3, 251-252.)—The authors have investigated a process proposed by Goske (ANALYST, 1910, 35, 162) for the estimation of husk in cocoa powders, and find that the results obtained by the method are neither concordant nor accurate. The sediments yielded by thirteen samples of commercial husk varied from 21·26 to 52·2 per cent. of the dry, fat-free substance, the average quantity being 36·96 per cent. Goske found much lower quantities. Results of the analyses of mixtures of cocoa powder with known quantities of husk show that the method does not afford reliable indication of the addition of less than 5 per cent. of husk, or of the amount of added husk actually present. W. P. S.

Note on the Detection of Cocoanut Oil in Chocolate. M. J. Wauters. (*Seventh Inter. Cong. App. Chem.*, 1909, Section VIII.c, 194.)—The evaporation to dryness in a strong current of air of the ethereal extract from a chocolate gives a residue having quite a different appearance when cocoanut oil is present from that of true cocoa butter. The addition of 10 per cent. of the former can be thus detected. The critical temperature of dissolution of cocoa butter in absolute alcohol is 78° C., while the figure for cocoanut oil is 34° to 35° C. H. F. E. H.

Analysis of Cod-Liver Oil by Means of its Miscibility Curve. E. Louïse. (*J. Pharm. Chim.*, 1911, 3, 377-385.)—The author has shown previously that a curve plotted from the results obtained in determinations of the miscibility points of oils with acetone is of use in ascertaining the quality of various oils (ANALYST, 1907, 32, 365) and of turpentine oil (ANALYST, 1910, 35, 322), and he now records the results found on the application of the method to cod-liver oil. Fifteen samples of colourless cod-liver oil gave almost parallel curves lying close together; the miscibility points of these oils, for quantities varying from 3 to 20 grms., were always above 0° C. The curves obtained with brown oils were of somewhat different form, and the miscibility points were all below 0° C., whilst the curves of light and amber-coloured oils lay between those of the colourless and brown oils. The miscibility curves indicate differences in the qualities of the oils more plainly than do the usual analytical constants. W. P. S.

Detection of Resin in Glazed Coffee. T. von Fellenberg. (*Mitt. Lebensmitteluntersuch. u. Hyg.*, 1911, 1, 301-302; through *Chem. Zentralbl.*, 1911, 1, 1253.)—The presence of resin on glazed coffee-berries cannot be detected directly by means of Storch's reaction (coloration with acetic anhydride and sulphuric acid), as coffee-berry oil itself gives a similar violet coloration. As, however, the substance in the coffee-berry oil which yields the reaction is an indifferent compound, whilst the resin reaction is due to the presence of abietic acid, the test may be used as follows: 10 grms. of the coffee are treated for a few minutes with 20 c.c. of ether, and the filtered ethereal extract is shaken with 3 c.c. of 5 per cent. sodium hydroxide solution. The aqueous solution will now contain the abietic acid, the acids of the coffee-berries, and traces of coffee-berry oil. The latter is removed by extracting the solution several times with ether, the solution being then acidified with hydrochloric acid, extracted with ether, the ethereal solution evaporated, and Storch's test applied to the residue obtained. Whilst pure shellac does not yield a coloration with the test, shellac glazes on coffee-berries will usually give a positive reaction, as the commercial shellac used for this purpose is almost invariably adulterated with resin (colophony). W. P. S.

Analysis of Commercial Dextrins. C. A. Browne and A. H. Bryan. (*Seventh Inter. Cong. App. Chem.*, 1909, Section V., 337-342.)—According to the method proposed by the authors, the percentages of dextrin and dextrose are calculated from the rotation and cupric-reducing power of the cold-water extract, taking the specific rotation of pure dextrin as 186, and the difference between the sum of these percentages and the dry organic solids of the extract is expressed as undetermined soluble organic matters. J. F. B.

Analysis of Honey. H. Witte. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 305-374.)—In continuation of a previous investigation on the composition and analysis of honey (ANALYST, 1910, **35**, 61) the results are now recorded of the analysis of forty-nine samples of pure honey, twenty-five samples of artificial honey, twelve samples of honey of doubtful purity, and nineteen samples of adulterated honey, these results, together with the behaviour of the samples towards various qualitative tests, being given in the form of eight separate tables. As the outcome of this investigation, the following limiting values for pure honey are suggested, and the reliability of certain tests is discussed: The quantity of water in honey should not exceed 20 per cent., and the acidity (calculated as formic acid) should lie between 0·06 and 1·2 per cent. The quantity of ash is generally from 0·1 to 0·35 per cent.; it may be as low as 0·065 per cent. The amounts of ash and acidity of a sample, however, afford little information as to the purity of the honey. The sucrose should not exceed 6 per cent., a larger quantity than this indicating that the honey is "unripe," that the bees have been fed on sucrose, or that the honey is adulterated. The author considers that "unripe" honey and that produced by sugar-feeding are not normal honeys. It may be mentioned that, at a suitable temperature, sucrose is inverted rapidly by certain honeys, and it is not possible to detect such inverted sugar by analysis. The quantity of nitrogenous substances in pure honey varies from 0·25 to 0·55 per cent., whilst artificial honey rarely contains more than 0·2 per cent. The catalase test and the *p*-phenylenediamine test proposed by Auzinger (ANALYST, 1910, **35**, 165, 259) do not always yield definite evidence as to the purity of a sample of honey, but, provided that the honey has not been strongly heated, the diastase test described by the same author is useful. Fiehe's reaction (ANALYST, 1908, **33**, 397) in conjunction with this diastase test is of great value in ascertaining the purity of a specimen of honey, but it must not be forgotten that artificial honeys occasionally give a negative reaction with Fiehe's test, and that honey which has been heated for a long time will yield a positive reaction. The faint coloration sometimes given by honey which has only been heated in the usual manner during its preparation cannot be mistaken for the cherry-red coloration yielded by artificial honey (invert sugar). In conclusion, the author recommends that all the data obtained during the analysis of a sample of honey ought to be considered together before an opinion is expressed as to the character of the honey. W. S. P.

Analysis of Honey, with Special Reference to the Nitrogenous Constituents. R. Lund. (*Mitt. Lebensmittelsunters. u. Hyg.*, 1911, **1**, 38-58; *Chem. Zentralbl.*, 1911, **1**, 1158-1159.)—As the result of the examination of a large number of samples of honey, the author concludes that in genuine honey about one-half of the total nitrogen exists in the form of protein and one-half as amino nitrogen, the quantity of ammonia nitrogen being negligible. Samples of genuine honey showed from 0·31 to 0·45 per cent. of total nitrogen, whilst artificial honeys contained only 0·106 to 0·20 per cent. For the estimation of the protein in honey, the author recommends a solution of 2 grms. of phosphotungstic acid in 20 grms. of dilute sulphuric acid (1 : 4) and 80 c.c. of water. Five c.c. of this reagent are added to 20 c.c. of a filtered 10 per cent. solution of honey, the mixture being diluted to 40 c.c. and stirred. The precipitate separates better in this way than when tannic acid is used, and the results are equivalent. Precipitation is effected in the test-tubes employed by Barth for the estimation of tannin in wine. The volume of the precipitate is read off after twenty-four hours, and in the case of natural honey it ranges from 0·6 to 2·7 c.c. Artificial honey gives either no precipitate or less than 0·5 c.c. If proteins have been added to artificial honey, differentiating tests, such as Bräutigam's, Ley's, and Fiehe's reactions, may be applied. J. F. B.

Volatile Acids of Honey. A. Heiduschka and G. Kaufmann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 375-378.)—The volatile acids occurring in honey do not consist wholly of formic acid; in some cases this acid constitutes only about one-half of the total volatile acids. The authors employed two methods for estimating the amount of formic acid in various samples of honey—namely, the mercuric chloride method (reduction to mercurous chloride), and the sulphuric acid method (measurement of the carbon monoxide produced). The former method yielded from 0·006 to 0·01 per cent. of formic acid, and the latter from 0·005 to 0·0073 per cent.; these results confirm the statements of other workers that the sulphuric acid method yields slightly lower results than does the mercuric chloride method. After saponification, larger quantities of volatile acids were obtained from the honeys, showing that a portion of these acids, including the formic acid, was present in the honeys in the form of esters. The total amount of formic acid (free and combined) found varied from 0·0118 to 0·0229 per cent. by the mercuric chloride method, and from 0·0096 to 0·0208 per cent. by the sulphuric acid method. W. P. S.

Notes on Linseed and other Oils. H. Ingle. (*J. Soc. Chem. Ind.*, 1911, **23**, 344.)—The author has examined the relationship between the iodine value (Wijs) specific gravity and the yield of hexabromides by Hehner and Mitchell's method (ANALYST, 1898, **23**, 310). One to 1·5 grms. are dissolved in a weighed flask in 40 c.c. of ether (sp. gr. 0·720) and 5 c.c. glacial acetic acid. Excess of bromine is run in, and after six hours the precipitate is collected on a tared paper, washed four times with 10 c.c. of ether, and the total weight of the precipitate determined after drying the residue still left in the flask, and that on the filter in the water oven. Samples of known purity gave values as follows:

Oil.	Iodine Value.	Sp. Gr. at 15° C.	Hexabromides. (1)	(2)	Melting-point °C.
Baltic oil	197·0	0·9357	48·1	47·5	140–5
Calcutta oil	185·0	0·9322	39·1	39·3	140–5
Dutch crushed ordinary ...	181·5	0·9322	36·9	—	140–3
English " " ...	185·0	0·9332	40·0	—	140–3
Plate oil	179·5	0·9315	35·3	33·7	140–5
Menhaden oil	182·0	0·9328	61·8	—	decomposes
Tung oil	168·0	—	nil		—

These results, with the exception of the melting-points, are not at all in agreement with Hehner and Mitchell (*loc. cit.*). The length of time of the stand with bromine and ether is immaterial. The following are some iodine values obtained: Baltic oils, 190 to 204 (good samples should not fall below 195); La Plata, 175 to 186 (the higher figure is now commoner than formerly); Black Sea oils, 176 to 182; North-American oils, 177 to 188 (very variable); Morocco, Dutch, and Turkish oils, 185 to 192. The Hübl method, modified by the author (ANALYST, 1902, 27, 279), gives similar values. Figures are given showing the lowering effect on the iodine and hexabromide values due to heating a sample of linseed oil for varying times at 195 to 200° C.

H. F. E. H.

Analyses of Evaporated Milk and Milk Products. E. Gudeman. (*Seventh Inter. Cong. App. Chem.*, 1909, Section VIII.c, 237.)—Butter fat in concentrated milk and in milk products is estimated as follows: 2·5 grms. of the milk product are dissolved or made into an emulsion with about 100 c.c. of water. Fat, proteins, and insoluble substances, such as starch, gums, fibre, etc., are precipitated with copper solution (Fehling), and the precipitate filtered, washed, and dried at a low temperature between filter paper. This is then extracted with ether, and the solution dried on the water-bath. The residue after ether extraction is used for the estimation of insoluble protein, the Kjeldahl digestion being complete in less than one hour. Sugar is determined in the filtrate from the copper sulphate precipitation. A preliminary extraction with ether is recommended for bodies rich in fat, such as milk chocolates and milk powders. Many substances containing sugars, starch, and cereals may be treated in this manner.

H. F. E. H.

Composition of Dried Milk Powders. A. Burr. (*Milchw. Zentralbl.*, 1911, 7, 118-113; through *Chem. Zentralbl.*, 1911, 1, 1234.)—The following results were obtained on the analysis of samples of dried milk preparations. For the estimation of the fat, the Rose-Gottlieb process, as modified by Richmond (ANALYST, 1908, 33, 389), and the Werner-Schmidt process, were found to be trustworthy, but the Adams process yielded low results. The factor 6·37 was employed for converting the nitrogen found by Kjeldahl's process into protein:

	Water.	Fat.	Proteins.	Lactose.	Ash.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Full-milk powder (37 samples) ...	1·20 to 10·04	22·89 to 32·41	23·79 to 34·09	31·63 to 45·13	5·48 to 7·54
Cream powder (22 samples) ...	0·92 „ 8·21	30·23 „ 53·52	—	—	—
Skimmed-milk powder (20 samples)	2·62 „ 13·88	0·63 „ 4·18	—	—	—
Whey powder (2 samples) ...	2·10 „ 2·20	1·63 „ 1·70	14·01 to 14·48	75·44 to 75·57	8·38 to 8·99

With the exception of the water, the figures are calculated on the dry substance. Only three of the so-called cream powders contained more than 50 per cent. of fat, and in many cases they did not differ essentially from full-milk powders. The author is of the opinion that a full-milk powder should contain not less than 25 per cent. of fat. Apart from the two whey powders, the preparations did not dissolve completely in water, and, as the powders undergo certain changes which affect their taste and solubility, the author considers that milk powders cannot be termed satisfactory preparations. The use of alkalis in the manufacture of the powders may be detected by testing their aqueous solution with litmus paper*; the test, however, is of use only when the powder is quite fresh, or has been kept out of contact with air, as the powder soon develops a high degree of acidity. W. P. S.

Polarimetric Estimation of Nicotine in Tobacco Extracts. J. von Degrazia. (*Fachl. Mitt. d. œsterr. Tabakregie*, 1910, 87–90; *Chem. Zentralbl.*, 1911, 1, 1085.)—The specific rotation of nicotine changes very appreciably with the temperature. According to the method proposed by the author, 30 grms. of tobacco extract are treated with 3·5 grms. of calcium oxide and 10 c.c. of water, and the mixture is distilled in a current of steam. When the distillate amounts to six times the quantity of tobacco extract taken, practically all the nicotine will have distilled over. The rotation of the distillate is then observed in a 200 mm. tube with a Lippich half-shadow polarimeter. The percentage of nicotine is calculated from the formula $P = \frac{a.G.f}{g}$, in which G = the weight of the distillate, g that of the tobacco extract, and f = a factor (the rotation constant) which varies according to the temperature and the rotation. Values of *f* determined for different temperatures and angles of rotation are given in the following table :

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* NOTE.—The use of rosolic acid is much to be preferred.

Temperature.	0·5°	1°	2°	3°	4°	5°
10° C ...	0·6365	0·6394	0·6405	0·6414	0·6419	0·6414
15° C ...	0·6315	0·6340	0·6349	0·6355	0·6354	0·6352
20° C ...	0·6264	0·6286	0·6293	0·6295	0·6288	0·6289

J. F. B.

New Method for the Estimation of Nicotine in Tobacco. J. von Degrazia. (*Fachl. Mitt. d. œsterr. Tabakregie*, 1910, 149–152; *Chem. Zentralbl.*, 1911, **1**, 1085–1086.)—Twenty grms. of finely powdered tobacco are treated in a flask of 300 c.c. capacity with 7 c.c. of potassium hydroxide solution (1 : 1), a few grms. of sodium chloride, and 130 c.c. of boiling concentrated brine. About 45 c.c. are rapidly distilled over without the use of steam, and then distillation is further continued in a current of steam, until 100 c.c. of distillate are obtained. Only with tobaccos very rich in nicotine is it necessary to distil off more. After the introduction of the current of steam, the direct heating of the flask should be interrupted for a time, to allow of the dilution of the residue. The distillate is weighed and polarised, the percentage of nicotine being calculated by the formula given in the preceding abstract. The results agree very closely with those obtained by Kissling's method (*cf.* ANALYST, 1911, pp. 106 and 143). J. F. B.

Estimation of Starch Syrup in Jams, etc. K. Beck and P. Lehmann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 393–405.)—Two processes are described, the first being intended for use in the case of jams, etc., which are not homogeneous, whilst the second is applicable to samples of a more uniform composition. The factors taken into account in the formulæ given are : The specific rotation of the inverted dry solids of starch syrup = +134·1°; the specific rotation of the inverted soluble substances of the jam = −21·5°; and starch syrup contains an average of 18 per cent. of water. The tables given in the official (German) methods for the analysis of wine are used for finding the quantity of soluble matters corresponding with the specific gravity of the jam solution.

Method I.—The amounts of soluble and insoluble substances are first estimated. One hundred grms. of the sample are mixed with 200 grms. of dilute alcohol (1 part of alcohol to 2 parts of water) of which the alcoholic strength, p, has been exactly found by taking the sp. gr. of its distillate, and the mixture is allowed to stand 24 hours, with occasional shaking. After filtering, 50 grms. of the filtrate are mixed with 25 c.c. of water, distilled, and the sp. gr. of this distillate is determined, giving the percentage quantity of alcohol now present, p_1; the percentages of alcohol, p and p_1, are expressed in parts by weight. The percentage quantity of insoluble substances, I, is then calculated from the formula

$$I = 100\left(3 - \frac{2p}{p_1}\right).$$

The residual solution in the distillation flask is now diluted to a volume of 50 c.c., its sp. gr. is determined, and the quantity of soluble substances is calculated from the sp. gr.; this quantity, multiplied by 3, gives the percentage quantity, t, of soluble substances in the sample. One-half the weight of the 50 c.c. of solution is now transferred to a 50 c.c. flask, 13 c.c. of water and 4 c.c. of 38 per cent. hydrochloric acid are added, and the mixture is heated to 68° C. After cooling, the solution is neutralised, 5 c.c. of basic lead acetate solution are added, the mixture is diluted to the mark with water, and filtered. Twenty-five c.c. of the filtrate are then treated with 2·5 c.c. of saturated sodium phosphate solution, filtered, and the rotation of the filtrate observed in a 220 mm. tube. The reading, when multiplied by 3, gives the specific rotation ($[\alpha]_D$) of the sample. The percentage quantity of starch syrup, y, is found from the formula

$$y = \tfrac{1}{6}(t + 4{\cdot}651\,[\alpha]_D).$$

Example.—Sp. gr. of the solution $= 1{\cdot}0502 = 13{\cdot}00$ grms. of soluble substances, and $13{\cdot}0 \times 3 = 39{\cdot}0$ per cent. of soluble substances (t). The observed rotation $= +2{\cdot}8°$; then $2{\cdot}8 \times 3 = 8{\cdot}4$, the specific rotation of the sample. By the above formula

$$y = \tfrac{1}{6}(39{\cdot}0 + 4{\cdot}651 \times 8{\cdot}4) = 13{\cdot}0 \text{ per cent. of starch syrup.}$$

This quantity is then multiplied by the factor $1 - (0{\cdot}0033 \times I)$, in order to correct it for the volume of the insoluble substances and to obtain the actual quantity of starch syrup present.

Method II.—Fifty grms. of the sample are mixed in a tared flask with 200 c.c. of 25 per cent. alcohol, 20 c.c. of basic lead acetate solution are added, and a further quantity of the dilute alcohol is added to make the total weight of the mixture 320 grms. After mixing and allowing to stand for 30 minutes, the mixture is filtered and the filtrate is collected in a weighed flask; the excess of lead is removed from the filtrate by treatment with hydrogen sulphide, and, if necessary, water is added to make up any loss in weight. After filtration, 50 c.c. of the filtrate are evaporated to expel the alcohol, the residual solution is diluted to a volume of 50 c.c., and its sp. gr. is determined, the corresponding quantity of soluble substances, E, being then calculated. Of this solution, 41·7 c.c. are transferred to a 50 c.c. flask, inverted as described above, and polarised in a 200 mm. tube (the treatment with sodium phosphate is not required). The percentage amount of starch syrup, y, is then found from the formula

$$y = E + 3P,$$

where P is the polarimeter reading.

Example.—Sp. gr. of the solution $= 1{\cdot}0271 = 7{\cdot}01$ E, and the rotation found $= +2{\cdot}6°$; then

$$y = 7{\cdot}01 + (3 \times 2{\cdot}6) = 14{\cdot}8 \text{ per cent. of starch syrup.}$$

This quantity may be corrected for the volume of the insoluble matters present as described previously.

W. P. S.

Detection of Prune Juice and Caramel in Vanilla Flavouring Extracts. W. Denis. (*J. Ind. Eng. Chem.*, 1911, 3, 254-255.)—The ordinary basic lead acetate test for caramel is quite worthless in the case of extracts containing more than 0·1 per cent. of vanillin; when more than this quantity is present, it is precipitated by the basic lead acetate, and carries down with it practically the whole of the caramel which may be in the extract. The following modification of the test has therefore been devised, and has been extended to include the detection of prune juice. Twenty-five c.c. of the extract are mixed with 50 c.c. of water, evaporated to a volume of about 20 c.c., and filtered. The filtrate, when cold, is extracted twice with ether, using 15 c.c. of the latter each time; the colour of the first ethereal extract should be noted, as the colouring matter of the vanilla bean is fairly soluble in ether, whilst caramel and prune juice are quite insoluble. The aqueous liquid is now transferred to a beaker, heated to remove dissolved ether, and treated, when cold, in a cylinder with 1 c.c. of basic lead acetate solution of sp. gr. 1·25. If caramel is present, the precipitate produced will be dark brown in colour, whilst the supernatant liquid will also be dark coloured. Five c.c. of glacial acetic acid are then added; in the presence of a pure extract, or of an extract coloured with caramel, the precipitate will be dissolved immediately. If prune juice has been used, a reddish-brown gelatinous mass will remain insoluble in the acetic acid. The colouring matter of sherry is said to be sometimes used to colour vanilla extract; this colouring matter gives a light-coloured precipitate with basic lead acetate, closely resembling in appearance the precipitate obtained from a pure vanilla extract, but differing from the latter in containing a body which is insoluble in acetic acid. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Germicidal Action of Arsenic and Antimony Compounds on Bacillus Typhosus. G. T. Morgan and E. A. Cooper. (*Seventh Int. Cong. App. Chem.*, 1909, Section IV A, 365-366.)—The Rideal-Walker test was used under standard conditions, employing a twenty-four hours' culture of the organism. The following are some of the results obtained:

Substance.	Co-efficient.	Dilutions Employed.
Phenol	1	1 in 100 killed in about 5 minutes.
Atoxyl (sodium salt)	Under 0·1	1 in 10 failed to kill in 15 minutes.
Potassium dicamphorylarsenate ...	0·25	1 in 75 ,, ,, ,,
Arrhenal	0·5	1 in 50 ,, ,, ,,
Potassium arsenate	0·2	1 in 20 ,, ,, ,,
Potassium arsenite	1·3 (approx.)	1 in 140 killed in about 5 minutes.
Diiodomethylarsenic acid	1·25	1 in 120 ,, ,, ,,
Triphenylantimonic hydroxynitrate	4·0 (approx.)	1 in 400 ,, ,, ,,

The quinquevalent arsenic compounds above, with one exception, are but feebly germicidal, and in that case the iodine confers the greater activity. In the presence of potassium arsenite the subcultures grew in an erratic manner, and the authors point out that mercuric chloride also behaves similarly unless a trace of sulphuretted hydrogen is added to combine with the carried-over salt, and so prevent the formation of a compound between the albumin of the germs and the mercury. It is

suggested that the toxicity of tervalent arsenic is due to a combination of this kind between the unsaturated arsenic and the organism, thus accounting for the irregularity in its action.
H. F. E. H.

Contribution to the Study of Chemical Disinfectants. Second Paper. S. Delépine. (*J. Soc. Chem. Ind.*, 1911, **23**, 334-343; *cf.* ANALYST, 1911, 69.)—The author reviews the work of the chief investigators concerning the action of chemical disinfectants, dealing first with Baxter on the action of various agents upon organic fluids that had undergone spontaneous putrefaction, and then with the same author's work on vaccine lymph, septic peritonitis, and glanders nodules. Of methods adopted since 1881, he describes Koch's use of pure cultures as test organisms acting in presence of large quantities of the disinfectant. This worker combined the use of the "thread" method with solid media, and was followed by Fraenkel and Behring, who substituted peptone bouillon for the solid medium in order to minimise the "carry over" danger. Krönig and Paul's "garnet" method is criticised, as are the modifications introduced by Sternberg, Wynter Blyth, and Rideal and Walker; and the author describes in detail the "suspension" and "thread" methods he has himself mostly employed.

The "suspension" method resembles in some respects that used by Baxter and Sternberg, and is used with such modifications as the occasion requires. Comparisons with phenol as the standard were almost always used, and in all cases it is easy to estimate accurately the proportion of bacteria, associated matter, and of disinfectant. No cotton-wool is employed, and the whole of the procedure is carried out in a small double-walled chamber, kept at 19° to 20° C. A mechanical rocker is employed (and illustrated) to keep the fluids in motion.

The Thread Method (*J. Roy. San. Inst.*, 1907, 28).—This differs from the former as regards the state of the test bacteria, and full details are given for the use of *B. coli communis* and *B. typhosus*. Unlike Rideal and Walker, the author does not express his results by means of a phenol co-efficient, but states exactly under what conditions the death of the test organisms occurred. In both the thread and suspension methods there is in each set of tests but one variant—the strength of the disinfecting solution; whereas Rideal and Walker have two—the degree of dilution and the time of exposure. The author gives various reasons for believing that either of his methods is preferable to the Rideal and Walker, and especially claims its superiority for testing disinfectants to be used on surfaces or articles contaminated with dry infectious products.

After discussing the objects of standardisation of disinfectants from the chemical and hygienic points of view, the author concludes: (1) That the use of naked bacteria is sometimes advantageous when the bactericidal powers of disinfectants, and not the actual hygienic practice, is under consideration, and that his suspension method is then preferable to the drop (or Rideal-Walker). (2) That the results obtained by these methods may be expressed by a figure indicating the bactericidal value of a disinfectant, carbolic acid being the standard; but that this figure indicates only a ratio obtained under conditions not necessarily occurring in practice, and that the term "co-efficient" should not be understood to indicate the hygienic value. (3) The hygienic value can be ascertained only by means of a series of tests in which practical conditions are represented as closely as possible. H. F. E. H.

Detection of Blood by Means of Leuco-Malachite Green. F. Michel. (*Chem. Zeit.*, 1911, **35**, 389-390.)—A reagent may be used for the peroxydase test in blood by dissolving 1 grm. of leuco-malachite green in 85 grms. of glacial acetic acid, and diluting the mixture with water to a volume of 250 c.c. One drop of the solution to be tested for blood is treated with 5 drops of the reagent and 2 drops of 1 per cent. hydrogen peroxide. If the drop of solution contains only as little as 0·002 mgrm. of blood, a green coloration will be obtained. W. P. S.

Estimation of Catalase in Milk. O. Laxa. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 417-420.)—An apparatus is described for measuring the quantity of oxygen liberated from hydrogen peroxide by the catalase present in milk. It consists of a graduated glass tube, the lower end of which is constricted to form a narrow jet, whilst the upper end is fitted with a tap. The capacity of the tube is 20 c.c. A mixture of the milk to be tested and hydrogen peroxide is prepared in the proportion of 15 c.c. of milk to 5 c.c. of 1 per cent. hydrogen peroxide solution, and the apparatus is filled with this mixture. After the lapse of a definite period of time, the volume of oxygen liberated by the action of the catalase is noted. Results of experiments with numerous samples of milk are given, showing that the apparatus yields concordant figures, and that the latter agree closely with those obtained by Koning's process (titration of the excess of hydrogen peroxide). The method described by Burri and Staub (ANALYST, 1909, **34**, 160) was found to yield unreliable results, probably owing to the liability of the agar cylinder to shrink and allow the gas to escape. W. P. S.

Estimation of Inorganic Phosphorus in Animal Tissues. A. C. Whittier. (*J. Ind. Eng. Chem.*, 1911, **3**, 248-250.)—The following method, due to Forbes (*Ohio Agr. Exp. Sta.*, *Bulletin* **215**, 481), is recommended: Extraction is accomplished by boiling the sample in ammonium sulphate solution, followed by repeated extractions with hot water. The determination is made by precipitating the inorganic phosphates with magnesia mixture and ammonia, followed by the regular gravimetric method for phosphorus on the dissolved magnesium precipitate. The method gives almost identical results with and without the application of heat to cold water extracts of flesh, and the results agree well with those obtained by the method of Emmet and Grindley (ANALYST, 1906, **31**, 159). Trowbridge and Stanley (ANALYST, 1910, **35**, 311) have pointed out that methods depending on the use of boiling water are faulty, because at this temperature organic compounds of phosphorus may be hydrolysed. The present author finds no evidence of this. Such hydrolysis as may occur on storage seems rather to depend on the action of enzymes and bacteria, and boiling arrests such action, and, in addition, gives rise to clear solutions, in which the subsequent precipitation is more complete. It is shown that Trowbridge and Stanley's determinations of organic phosphorus in uncoagulated extracts by the barium method are too high, and this is traced to the passage of barium phosphate through the filter. G. C. J.

Experiments on the Antiseptic Properties of Sulphurous Acid, Sulphites, and Complex Compounds of Sulphurous Acid. E. Hailer. (*Arbeit. Kaiserl. Gesundheitsamte*, 1911, **36**, 297-340; through *Chem. Zentralbl.*, 1911, 1, 1309-1311.)—The relative concentrations of sulphurous acid necessary to destroy moulds, yeasts, and bacteria, were in the order 5, 4, and 1, respectively. The addition of dextrose to the media containing the organisms decreased the action of the sulphurous acid. The latter has a more powerful action at a temperature of 37° C. than it has at 22° C. Sodium sulphite, in the concentrations employed in the experiments, was without effect on the organisms, whilst sodium bisulphite was less effective than an equivalent quantity of sulphurous acid. All three kinds of organisms were equally resistant to the action of phenol, a $\frac{1}{35}$-molar solution being required to kill them, whilst moulds were destroyed by a $\frac{1}{150}$-molar solution of sulphur dioxide, yeasts by a $\frac{1}{186}$-molar solution, and bacteria by a $\frac{1}{750}$-molar solution. A much higher phenol concentration is, therefore, required to kill the organisms than is necessary in the case of sulphurous acid. The compounds of formaldehyde and acetaldehyde with sodium bisulphite, even in high concentrations, do not affect moulds, but the corresponding compounds of acetone and dextrose have some action. The dextrose compound has the most effect on bacteria, then follows the acetone compound, and next the acetaldehyde compound. The quantity of sulphurous acid necessary for the effective preservation of a substance depends to a great extent on the composition of the latter; and, without taking into account its physiological action, sulphurous acid, when used as a preservative, possesses the disadvantage that it is volatile and is also readily oxidisable. As regards its employment in the case of dried fruits, experiments showed that there was no need for its use when the fruits are properly dried. The alleged necessity for the use of sulphurous acid in certain classes of wine is being investigated. W. P. S.

Estimation of Hexamethylenetetramine (Urotropine) in Urine. F. Schröter. (*Archiv. exper. Pathol. Pharm.*, 1911, **64**, 161-166; through *Chem. Zentralbl.*, 1911, **1**, 1257.)—As inaccurate results are obtained by estimating the nitrogen in the mercuric chloride precipitate obtained from urine containing hexamethylenetetramine, owing to the occlusion of uric acid in the precipitate, the following method, in which the precipitate is treated for the removal of the uric acid, is recommended. One hundred c.c. of the urine are mixed with 10 c.c. of 25 per cent. acetic acid, and treated with 120 c.c. of saturated mercuric chloride solution. After the lapse of twelve hours, the precipitate is collected on a filter, then digested on the water-bath with 15 c.c. of concentrated sodium chloride solution, and again filtered; the hexamethylenetetramine-mercuric chloride compound dissolves in the sodium chloride solution, and, after the mercury has been removed by the addition of sodium hydroxide, the nitrogen in the solution is estimated by Kjeldahl's process. The amount of nitrogen found is then calculated into hexamethylenetetramine.

W. P. S.

Bacteriology of Water: Its Present Position. P. F. Frankland. (*J. Soc. Chem. Ind.*, 1911, **23**, 319-334.)—The author reviews the present position of the knowledge on this subject, special mention being made of his own work and the various recent reports of Houston to the Metropolitan Water Board. A short historical summary is followed by numerous tables dealing with the biochemical reactions of *B. typhosus*, *B. coli*, and streptococci, and the rate of disappearance of the former from raw river and stored waters. An account is given with tables of recent bacteriological methods as applied to testing the purity of waters which have been treated by processes other than storage and sand filtration. These include mechanical filters, Clarke's process of softening, lime and alumino-ferric, ozone, bleaching powder and ultra-violet light. Illustrating the extremely laborious nature of the work done by Houston, a description in detail is given of one investigation involving the examination of 156 samples of raw river water containing 135,687,500 bacteria. From these 7,329 selected colonies were subcultured and fully examined for behaviour characteristic of *B. typhosus*. Not one of all these colonies agreed in all respects with any of the thirty-two known strains of this organism which were simultaneously examined. In another series, out of 5,451 colonies selected for special study from raw river water, one consisted of bacteria which closely resembled the typhoid bacillus.

The work of McConkey and Hill and Savage on *B. coli communis* is reviewed, the labour expended on "this now classical microbe" having been of wider scientific interest than its mere hygienic significance. The position taken up as long ago as 1886 by the author is vindicated with regard to the storage of water as effecting its purification, for, notwithstanding the great advances in the technique of typhoid-testing, the results obtained recently by Houston are essentially similar to those obtained by the author fifteen years previously.

The author fully endorses all the advantages which Houston claims are secured by the storage of surface waters.

In the final conclusion it is stated that no very surprising novelties have been brought to light since 1895. By that date all the salient features in the bacteriology of water had been mapped out, and recent work has filled in details and confirmed previous results broadly established. The all-important questions for the water-examiner remain the same: (1) Has the water been contaminated with the excreta of man? and (2) if so contaminated, has the water been since subjected to conditions sufficient to insure the destruction or removal of the pathogenic organisms which may so have been introduced? No categorical answer is yet possible to the first of these questions, because no organism absolutely characteristic of human excreta is yet known. *B. coli communis* is characteristic of fæcal matter in general, and all surface water must be contaminated more or less with this organism.

The second question also requires a qualified answer. If the water is free from *B. coli* in a large volume, it may be concluded that *a fortiori* it is free from typhoid. But should *B. coli* be found in a small volume, it does not prove that the water may at any time be liable to contain typhoid bacilli, because the presence of *B. coli* does not prove that the water has been contaminated by man. It is easier to exculpate a water than to incriminate it, but even the absence of *B. coli* from a large volume does not prove its safety in view of the recent remarkable revelation as to people who are typhoid-carriers, and who by discharging typhoid bacilli in their urine may contaminate water without causing it at the same time to receive any *B. coli*. The author deprecates the adoption of any hard-and-fast standard of purity such as the now popular "no typical *B. coli* in 100 c.c.," and also the idea that the chemical examination of drinking water has been superseded by the bacteriological, and asserts that the latter, while more useful for following the variation in purity of a known water may, if used without chemical analysis in a water of unknown origin, lead to erroneous conclusions. H. F. E. H.

ORGANIC ANALYSIS.

Use of Metallic Potassium in the Estimation of Halogens in Benzene Derivatives. C. H. Maryott. (*Amer. J. Sci.*, 1910, **30**, 378-380; through *Chem. Zentralbl.*, 1911, **1**, 1246.)—From 0·1 to 0·4 grm. of the substance is placed in a flask, 15 c.c. of a mixture of 1 part of alcohol with 2 parts of thiophene-free benzene are added, the flask is attached to a reflux condenser, and about ten times the amount of potassium required by theory is introduced gradually. After the contents of the flask have been heated and the reaction is completed, a further quantity of 2 c.c. of alcohol is added, and the heating is continued until the whole of the potassium has disappeared. The halogen in the resulting solution is then estimated in the usual way. The method yielded trustworthy results in the case of such substances as chlorobenzene, bromobenzene, hexachlorobenzene, and *p*-chloraniline. When metallic sodium was used as recommended by Stepanow (ANALYST, 1907, **32**, 52), only 84 per cent. of the quantity of halogen present was obtained. W. P. S.

Caoutchouc Nitrosite and its Analytical Application. P. Alexander. (*Zeitsch. angew. Chem.*, 1911, **24**, 680-687.)—The author has previously recorded the production of carbon dioxide during the preparation of caoutchouc nitrosite and has analysed the products obtained, suggesting the formula $C_9H_{12}O_6N_2$, instead of Harries' formula $(C_{10}H_{15}O_7N_3)_2$ for the "nitrosite-c." In the present paper he confirms his observations and defends his views against certain criticisms which have been made. He admits that the amount of carbon dioxide formed varies with the conditions under which the nitrosite is prepared, depending especially on the time and temperature of the treatment; but if care be taken to collect all the carbon dioxide produced, a quantity representing 86 per cent. of that calculated for the splitting off of 1 molecule of carbon dioxide from a body of C_{10} constitution may be found. The greater portion of this is evolved by secondary oxidation changes during the later stages of the reaction, but in any case there is always sufficient evolution of carbon dioxide to exclude the possibility of simple nitrosite formation quantitatively with the production of a nitrosite of C_{10} formula. The author concedes, however, that with very different varieties of caoutchouc the quantity of crude nitrosite obtained by the full action of nitrous vapours corresponds very closely with that calculated from Harries' formula. But this in no way proves the correctness of that formula for a purified derivative, but merely that the loss of carbon dioxide is approximately compensated by the fixation of oxygen in the residue. Dealing with the question whether the nitrosite reaction may be legitimately used for the quantitative estimation of caoutchouc, the author decides that if the conditions prescribed by Harries be accurately followed, Harries' calculation-factor $\frac{136}{284}$ may certainly be accepted as an empirical value, not, however, expressing the actual quantitative production of his "nitrosite-*c*," for which it was originally intended. For the estimation of caoutchouc in vulcanised products, however, the author cannot recommend the nitrosite method unreservedly, although in many cases he uses it for the estimation of the coefficient of vulcanisation with satisfactory results. It possesses a great advantage in enabling the analyst to eliminate all mineral loading materials, by dissolving the nitrosite in acetone and filtering the solution (*cf.* ANALYST, 1911, 158). J. F. B.

Sources of Error in the Folin Method of Estimating Creatinine. A. E. Taylor. (*J. Biol. Chem.*, 1911, **9**, 19-20.)—Personal equation, the absence of a standard light, and the presence of urinary pigment and bile are disturbing factors in this estimation.

Study of the Velocity of Detonation. A. M. Comey. (*Seventh Inter. Cong. App. Chem.*, 1909, Section III, B, 28.)—The velocities of detonation of American "straight" dynamites—*i.e.*, nitroglycerol absorbed in an active dope, gelatine dynamites, "ammonia" dynamites, and nitroglycerol in tubes, are determined by Bichel's well-known method and by the method of Dautriche (*Comptes rend.*, **143**, 641, and **144**, 1030). By the Dautriche method, applied to a row of sticks of dynamite rolled in paper with their ends touching, and detonated at one end, it was found that the velocity was constant for high-grade dynamites (*e.g.*, 5,803 to 5,793 metres per second for dynamite containing 60 per cent. nitroglycerol), increased from the first to the last stick for intermediate grades (4,000 to 4,356 for 33 per cent.), and tended to uniformity again for the lowest grades (3,144 to 3,200 for 27 per cent.). With blasting gelatine the velocity increased rapidly from the point of detonation, reaching a maximum in about 4 inches. The velocity of transmission through air from one stick to another was found generally to vary inversely as the distance, up to a maximum, according to the size of the primer. O. E. M.

Estimation of Essential Oils. C. D. Howard. (*J. Ind. Eng. Chem.*, 1911, **3**, 252.)—It is admitted that the method described previously by the author (ANALYST, 1908, **33**, 236), sometimes gives inaccurate results in the case of certain oils, although it has proved to be of use with such oils as lemon, orange, wintergreen, and peppermint. A modification of the method, has, however, been found to be trustworthy for the analysis of most varieties of essential oil preparations; it involves the application of the principle that when an ethereal solution of an essential oil is evaporated rapidly there is no appreciable loss of oil. The procedure is as follows: Twenty c.c. of the essence in which the essential oil is to be estimated are mixed with 50 c.c. of water and 2 drops of concentrated hydrochloric acid, and shaken out with three successive quantities (15 c.c., 10 c.c., and 5 c.c. of ether). The combined ethereal extracts are washed once with 10 c.c. of water saturated with ether, and then transferred to an ordinary Babcock milk-bottle. The latter is immersed in hot water and the neck is connected with a filter-pump; when most of the ether has evaporated, the bottle is shaken violently and immersed for a few seconds in boiling water until the ether has been completely expelled. Cold water is then added, the contents of the bottle are submitted to centrifugal action, and the volume of the oil is read. W. P. S.

Essential Oil of "False Camphor-Wood"; The Occurrence of Myrtenal and d-Perilla Aldehyde in Nature. F. W. Semmler and B. Zaar. (*Ber.*, 1911, **44**, 815-819.)—"False camphor-wood," of unknown botanical origin, yielded 2.06 per cent. of an essential oil having the constants: $d_{15} = 0{\cdot}958$; $[n]_{D20^\circ} = 1{\cdot}49695$; $[\alpha]_D = +98^\circ 10'$, soluble in 2·5 vols., and over, of 70 per cent. alcohol. This oil, on extraction with sodium bisulphite, yielded up to 75 per cent. of an aldehyde, $C_{10}H_{14}O$. The authors have isolated from the oil two aldehydes of the above formula. The main constituent is extracted by means of sodium bisulphite and is the dextrorotatory isomeride of the perilla aldehyde previously described. The second aldehyde is extracted by means of sodium sulphite, and is identical with myrtenal, hitherto obtained only synthetically. The oil contains, in addition, *d*-limonene and considerable quantities of cineol. Perilla aldehyde has a monocyclic constitution, and myrtenal a bicyclic. They stand, to one another, in the same relationship as limonene stands to pinene. J. F. B.

Analysis of Gelatine. J. Herold. (*Chem. Zeit.*, 1911, **35**, 93-94.)—The melting-points are taken of a 20 per cent. jelly prepared from the sample and of a 20 per cent. jelly made from a mixture of equal parts of pure gelatine and glutose (prepared by heating gelatine and ammonia in a sealed tube at a temperature of 100° C.). The percentage of glutin (x) in the sample is then calculated from the equation $x = 68{\cdot}3a$, where a is the difference in the melting-point.

Glucoside Reactions: Convallarin and Convallamarin. C. Reichard. (*Pharm. Zentral.-h.*, 1911, **52**, 183-188.)—These two glucosides occur in the roots and leaves of *Convallaria majalis*; convallamarin has a similar physiological action to that of digitalin, whilst convallarin acts solely as a purgative. The most characteristic reaction for differentiating the two substances is that obtained with sodium iodate; a mixture of convallarin and sodium iodate when dried yields a white residue which is not affected by hydrochloric acid, whilst the residue obtained in the case of convallamarin consists of a moist amorphous mass which is coloured yellow on the addition of hydrochloric acid. Reactions with numerous other reagents are described.

Estimation of Glycerol. E. Probeck. (*J. Ind. Eng. Chem.*, 1911, **3**, 253-254.)—From his experience with the acetin process as applied to the estimation of glycerol in crude glycerol, the author is of the opinion that the results obtained are from 0·2 to 0·5 per cent. too low. The loss is not due to the presence of impurities, but to the manipulations introducing sources of error. The bichromate method is preferable in the absence of acrolein and other volatile aldehydes; if aldehydes are present, they must be removed before the addition of the bichromate. Instead of removing chlorine and proteins from the crude glycerol previous to the oxidation, the author prefers to apply the bichromate process directly to the glycerol and to make a correction for the amount of chloride, etc. For this purpose, a quantity of the glycerol is neutralised and evaporated at a temperature of 160° C., and the residue is oxidised with bichromate. The quantity of bichromate thus required is deducted from that used for the total oxidation.

W. P. S.

Estimation of Indigotin in the Presence of Starch. W. Thomson. (*J. Soc. Dyers*, 1911, **27**, 49-51.)—In the case of mixtures consisting of synthetic indigo and starch, Rawson's permanganate process (with salt precipitation of the sodium indigotindisulphonate) yields low results. After treating the sample with 4 per cent. hydrochloric acid in order to hydrolyse the starch, and washing away the sugars formed, the process gives better results. Bloxam's tetrasulphonate process yields uniform results.

Estimation of the Acidity of Fats and Oils, particularly Lubricating Oils. H. Loebell. (*Chem. Zeit.*, 1911, **35**, 276-277.)—A mixture of alcohol and benzene is recommended as a solvent for the oil, the end-point of the titration being thus rendered sharp.

Estimation of the Saponification Value of Oils, Fats, and Waxes. Use of Propyl Alcohol. L. W. Winkler. (*Zeitsch. angew. Chem.*, 1911, **24**, 636-638.)—The employment of propyl alcohol in place of ethyl alcohol is recommended in the preparation of the alcoholic potassium hydroxide solution used in estimating the saponification values of fats and waxes; it may also be used as the solvent for the fat or wax in the estimation of the acid value. Warm propyl alcohol is a better solvent for fats than is ethyl alcohol, and, on account of its relatively higher boiling-point, the saponification of even beeswax is completed after ten minutes' boiling.

W. P. S.

Some Constants of Mutton-Bird Oil. L. H. Smith. (*J. Soc. Chem. Ind.*, 1911, **30**, 405.)—The oil carried in the stomach of the mutton-bird (*Aestrelata lessoni*), a petrel peculiar to the Antarctic regions, has the following characters: Sp. gr., 0·8819 to 0·8858 at 15·5° C.; saponification value, 125·9; iodine value (Wijs), 71 per cent.; unsaponifiable matters (alcohols), 36·88 per cent.; m.-pt. of alcohols, 30·5° to 31·5° C. It yields no glycerol, and is therefore not a glyceride but a liquid wax. At 0° C. it solidifies to a translucent mass.

The body-fat of the bird is of a soft consistency and has the following characters: Free fatty acids, 3·5 to 4·3 per cent. (as oleic acid); iodine value (Wijs), 89·1 per cent.; unsaponifiable matter, 1·76 to 2·0 per cent.; sp. gr., 0·9351 to 0·9380 at 15·5° C.; titre test, 29·4° C.

A. R. T.

Detection of Animal Sizing in Paper. Differentiation of Gelatin and Casein. C. Levi. (*Papierfabr.*, 1911, **9**, 365-367; through *J. Soc. Chem. Ind.*, 1911, **30**, 484.)—For the detection of animal size in paper, whether gelatin or casein, the author recommends the biuret reaction. A fragment of the paper is steeped for a few minutes in a 2 per cent. solution of copper sulphate, and is then treated with a

5 per cent. solution of sodium hydroxide, the latter being preferably dropped on the paper. If gelatin or casein be present, a violet coloration is immediately produced. For distinguishing between gelatin and casein in a paper found to contain animal size, the author recommends the xanthoprotein reaction, which is shown by casein, but not by gelatin. A fragment of the paper is moistened with a drop of concentrated nitric acid, and if casein be present an intense yellow stain is produced. This stain turns brown with caustic soda and orange with ammonia. The presence of mechanical wood-fibre vitiates this test, since lignin is also stained with nitric acid. In the case of papers coated with casein the coating may be scraped off, or in cases where the paper cannot be tested direct the casein may be extracted with borax or alkali, precipitated with acetic acid, and the dried precipitate tested with nitric acid or Millon's reagent.

Lubricating Oils for Motor-Cars and Gas-Engines. F. Schwarz and H. Schlüter. (*Chem. Zeit.*, 1911, **35**, 413-415.)—Oils for lubricating the cylinders of explosion motors must not be too viscous, nor have too high an ignition point, and it is essential that the portion which is inevitably consumed in the explosion should not contain too much hydrocarbon to be burned up by the available oxygen. If any carbonaceous residue is left, the gases escaping from the motor cylinders will have a blue-grey colour and an unpleasant odour. For this reason, any admixture of fatty oils to cylinder oils is now, as a rule, avoided. Fractionation of a lubricating oil by means of acetone into lighter and heavier portions will probably afford a means of ascertaining the liability of a cylinder lubricating oil to emit malodorous vapours in use. Thus, by practical tests it was found that the fraction insoluble in acetone produced only grey-white vapour, with a faint, not unpleasant, odour, whereas the original oil and the portions soluble in acetone emitted dirty grey vapours, with the well-known unpleasant odour. The following results are typical of those thus obtained:

	Per Cent. by Weight.	Colour in 15-Mm. Tube.	Sp. Gr. at $\frac{15^\circ}{4^\circ}$ C.	Viscosity, Engler (Water at 20° C. = 1).		Ratio Viscosity, $\frac{20^\circ}{50^\circ}$ C.	Flash-Point (Open Test).	Ignition Point.	Insoluble in Petroleum Spirit after 50 Hours at 50° C.
				at 20° C.	at 50° C.		° C.	° C.	Per Cent.
Motor-car oil	—	Light brown	0·9159	37·7	5·9	6·4	208	259	0·31
Insoluble in acetone	66	Ditto, slightly turbid	0·8959	43·6	6·9	6·3	215	257	0·04
Soluble in acetone, first extract	14	Light brown	0·9657	48·4	6·3	7·7	198	high 241	1·45
Ditto, second extract	12	Ditto	0·9552	43·6	5·7	7·7	191	237	—
Ditto, third extract	8	Brownish-yellow	0·9471	40·1	5·5	7·3	196	246	—

The petroleum spirit used for the estimation of the substances insoluble in petroleum spirit had a sp. gr. of 0·6324 at 15° C., and 93 per cent. passed over between 30° and 50° C. In the author's opinion, the most suitable lubricants for the cylinders of motor-cars are pure mineral oils, with a viscosity of 6 to 13 Engler's degrees at 50° C. (seldom greater). The proportion of paraffin wax should also be low, so as to prevent congealing in winter. Results obtained with representative motor and gas-engine lubricating oils on the market are shown in the following table, the practical tests having been made with motors of different strength:

	Viscosity, Engler (Water at 20°=1).		Sp. Gr. at 15° C. (Water at 4°=1).	Cold Test.	Flash-Point (Pensky-Marten).	Composition.	Behaviour in Practice.
	20° C.	50° C.					
				° C.	° C.		
MOTOR-CAR OILS:							
(a) *Of consistency of machine oil*—							
1	25·0	5·1	0·9077	+5 to 0	193	Pure mineral oil	Satisfactory as cylinder oil
2	68·8	9·6	—	—	224	Ditto	Ditto
(b) *Of medium and high viscosity*—							
3	about 128·0	8·3	0·9078	+15 to +10	65	Ditto	Satisfactory
4	—	9·6	0·8889	—	206	Contained fat	Deposit formed in cylinder
5	—	15·9	0·8996	Semi-solid at 0°	215	Pure mineral oil	Good as cylinder oil
6	—	36·0	0·9011	Ditto	238	Contained fat	Deposit formed in cylinder
GAS-MOTOR OILS:							
7	12·4	3·44	0·9035	—	211	Fat present	Satisfactory
8	70·1	7·8	0·9050	0 to −2·8	202	Mineral oil +2% fat	Unsatisfactory

Gas-engine oils have similar properties to those of motor oils. In one instance a sample gave a residue in the cylinders of the gas-engine, although in other respects it agreed with samples that were satisfactory. The tar value and tar-forming value were low, and the cause was not detected. The formation of a residue, however, is not always to be attributed to the lubricating oil. Thus, the use of coke-furnace gas which has been insufficiently freed from tar oil may lead to the deposition of a tarry residue in the cylinders.

C. A. M.

"Hughes' Reaction" of Potassium Iodide on Paper, and its Bearing on the Question of Acidity. J. Strachan. (*Chem. News*, 1911, **103**, 193-195.)—The author has studied "Hughes' reaction," which is observed when sized paper is moistened with a 20 per cent. solution of potassium iodide and exposed to the action of air free from acid or ammonia vapours, for an hour. Iodine is liberated, which produces a stain (blue when starch is present), more or less intense. The author considers that the occurrence of this reaction is due to the action of the undecomposed alum in the paper, in virtue of its acidity, upon the potassium iodide, and he

considers the presence of sufficient alum to give an intense coloration, according to Hughes' reaction, to be undesirable from the point of view of the permanence of the paper.
J. F. B.

Researches on the Quantitative Analysis of Pentoses and Pentosans, and its Practical Applications. R. Adan. (*Seventh Inter. Cong. App. Chem.*, 1909, Sect. VIII.c, 194-204.)—The gravimetric method of estimating furfural as phenylhydrazone, in the author's hands gave results with pure furfural which are accurate when calculated as xylose or arabinose; but the process is difficult and tedious, and requires a slight but varying correction for solubility of the hydrazone in the washing water. The phloroglucide method was generally employed, and gave results showing a maximum error of 1·5 per cent. when working on pure furfural. The proportion of phloroglucinol to be added to the distillate is double that of the furfural expected. The total volume of the distillate is then made up to 400 c.c. with HCl (sp. gr. 1·6), stirred, allowed to stand until the following day, collected on an asbestos filter, washed with 150 c.c. water, and dried at 98° C. for four hours. A sliding-scale correction has to be employed for the calculation of the result, which is given by Tollens and Gröber. The author has tried a method of estimating the furfural by adding excess of phenylhydrazine, and then measuring the nitrogen evolved from the uncombined phenylhydrazine in presence of copper sulphate, and thus by difference determining the amount required to combine with the furfural; but this, though applicable to pure solutions of phenylhydrazine, does not work well in presence of sodium chloride, and had to be abandoned.

In order to investigate the percentage of shell in cocoa-powder, the pentosans in both nibs and shell were estimated, using the distillation method of Tollens, and precipitating the furfural with phloroglucinol as described by König, "Untersuchung Landwirthschaftliche u. Gewerbliche Stoffe," 3rd ed., 1906. Two to 2·5 grms. were finely powdered and defatted. It was found from the mean of all analyses that 100 grms. of entire cocoa-bean gave 45 grms. of dry fat-free material, or 75 grms. of cocoa with 30 per cent. fat.

AVERAGE ANALYSIS OF SHELLED COCOA GRAINS.

Moisture.	Fat.	Starch.	Ash.	Cellulose.	Pentosans.			
					Grains.		Shells.	
					Initial Substance.	Powder with 30% Fat.	Dry Substance.	Substance with 10% Moisture.
6·43	44·44	8·22	4·00	4·78	1·53	2·47	9·96	8·21

AVERAGE COMPOSITION OF SIX COCOA POWDERS.

Moisture.	Fat.	Ash.	Cellulose.	Pentosans.	
				Initial Substance.	Powder with 30% Fat.
0·88	53·86	3·33	2·95	1·61	3·40

The wide variation between the percentage of pentosans in cocoa grains and that in shells—viz., 1·53 and 9·96—can be made the basis of an estimation of the quantity of the latter present in the finished article. H. F. E. H.

Technical Analysis of Petroleum Asphalt. D. Lohmann. (*Chem. Zeit.*, 1911, **35**, 357-358.)—The author claims that the analytical side of the asphalt industry is more fully developed in America than in Europe, and gives a blank form of the determinations usually made in this connection. The "penetration" test is made with Dow's apparatus, in which the penetration of a standard needle loaded with a standard weight is measured in tenths of a mm. at different temperatures. The penetration is an indication of the hardness, and to a certain extent of the ductility. The fixed carbon is determined by driving off the volatile matters in a covered crucible of standard dimensions with a standard flame. The sulphur is estimated in the calorimetric bomb of Mahler with oxygen under a pressure of 25 atmospheres, by absorption of the gases of combustion. The ductility is measured in an apparatus devised by Dow and Smith. A piece of asphalt, cast in a mould, is tested at a temperature of 77° F. by fixing one end and drawing out the other horizontally until the thread breaks. The test is performed under water, the speed of drawing is 5 cm. per minute, and the length of the thread is measured in cm. The ductility and penetration tests are the most important factors in the technical valuation of asphalt. A material of moderate hardness and inferior ductility would be quite unsuitable for street paving, at any rate if used alone. An asphalt of high ductility generally shows the greatest differences in the penetration tests at different temperatures. It is generally insisted that a petroleum asphalt should consist of nearly pure bitumen, soluble in carbon bisulphide up to a fraction of 1 per cent., and soluble within a few per cent. in carbon tetrachloride; thus it differs considerably from most natural asphalts. The solubility in petroleum spirit of sp. gr. 0·700 affords an indication whether a petroleum asphalt has suffered considerable decomposition during distillation, decomposed samples being less soluble in this spirit. The solubility, for the same reason, also bears a relationship to the ductility and the alteration of hardness with temperature. The percentage of sulphur appears to have no influence on any of the technically valuable properties. J. F. B.

Action of Iodine on Phenols and their Volumetric Determination. (Part I.). J. M. Wilkie. (*J. Soc. Chem. Ind.*, 1911, **30**, 398-402.)—The author reviews the various methods which have been proposed for the determination of phenol, salicylic acid, and β-naphthol by means of standard bromine and iodine solutions. As regards Messinger and Vortmann's method (ANALYST, 1891, **16**, 75), it is pointed out that good results are only obtainable when the proper proportions of the phenol, alkali, and iodine are present, and details of the method of working are given. Sodium carbonate is recommended as the alkali (in the cases of phenol and salicylic acid) instead of caustic soda. In the case of phenol and salts of salicylic acid, again, it is shown that the products of the action of iodine are two—namely, tri-iodophenol after a short period (five minutes), and, on long standing in contact with the reagent, tetra-iodo-diphenoquinone. With β-naphthol, a practically quantitative yield of 1·2 iodo-oxy-naphthalene is obtained when its aqueous solution is treated with $\frac{N}{10}$ iodine in the cold. With more sodium hydroxide than is necessary to form the sodium salt, the crystalline product is not produced, but the green amorphous substance described by Messinger and Vortmann (*loc. cit.*).

A. R. T.

Sensitive Test for Phenol and Salicylic Acid. J. M. Wilkie. (*J. Soc. Chem. Ind.*, 1911, **30**, 402-403.)—Although the sensitiveness of the tribromophenol method for these substances is limited by the considerable solubility of this derivative, as pointed out by Allen ("Com. Org. Analysis," vol. ii., part 2, p. 244) and confirmed by the author, the tri-iodo-phenol precipitates are not so soluble, and they thus provide a more delicate test for the presence of phenol and salicylic acid. The limit of sensitiveness of the reaction in the case of bromine-water is about 1 in 60,000; while in the case of iodine solution a precipitate is produced after one hour with solutions of salicylic acid of about 1 in 870,000 dilution, and of about 1 in 1,250,000 for phenol. The substance to be tested is treated with $\frac{N}{10}$ iodine and $\frac{N}{10}$ sodium carbonate, and allowed to stand for five minutes, when a slight excess of $\frac{N}{1}$ sulphuric acid is added. Amounts of phenol equal to 2 and 4 parts per million show the crystalline precipitate within five minutes, and 1 per million in about one hour, after the addition of the acid. The acid should be added within an hour of the commencement of the reaction, or a negative result may be obtained in the presence of the substances sought.

A. R. T.

Stability Tests of Smokeless Powder. G. W. Patterson. (*Seventh Inter. Cong. App. Chem.*, 1909, Section III.B, 99.)—The paper relates entirely to single base powders. The author considers it impossible to predict the life of a powder; if it shows a present wide margin of safety it is relatively safe. He describes modified forms of the German 135° C., and Vieille 18·5° C. tests, and gives details of his surveillance test. This consists in heating a standard weight of powder, about 45 grms., in an 8-oz. glass-stoppered bottle at 65·5° C., until red fumes appear. The number of days required is the index of stability. At 80° C. the time required is roughly a quarter of that at 61·5°.

O. E. M.

Experiments on the Precipitation of Proteins. A. J. J. Vandevelde. (*Bull. Soc. Chim. Belg.*, 1911, **25**, 166-173.)—By fractionally precipitating the proteins of milk by means of varying quantities of acetone, methyl alcohol, and ethyl alcohol in the presence of acetic acid, and then estimating the amounts of proteins in solutions of the precipitates, the author shows that the treatment to some extent transforms the proteins from one form to the other. Similar results were obtained in the case of beef serum, egg albumin, etc.

Influence of Certain Compounds upon the Colour Reactions of Strychnine. E. Mameli. (*Gazz. Chim. Ital.*, 1911, **41**, 329-334.)—The characteristic colour reactions given by a sulphuric acid solution of strychnine with potassium bichromate and with ammonium vanadate are masked completely by certain substances which include phenol, anethol, phenetole, β-naphthol, β-naphthyl salicylate, benzonaphthol, and dulcine. Acetonylacetone and salol have the same effect upon the bichromate reaction, but only when present in considerable quantity do they interfere with the vanadate reaction, causing the coloration to become green. In the presence of a large excess of strychnine the colour is violet instead of green. Aniline, acetic acid, tartaric acid, stovaine, and urotropine do not interfere with the vanadate reaction, but weaken, and, if present in sufficient quantity, prevent the bichromate reaction. Zinc lactate and orthotoluidine inhibit the bichromate reaction and weaken the vanadate reaction, which is also rendered fugitive by the presence of quinoline and terpene. Otto's (bichromate) reaction is not materially affected by piperonal, lecithin, and papain, but the coloration in the vanadate reaction is modified and rendered fugitive by their presence. Antipyrin, aspirin, menthol, analgin, and salicylic acid mask the bichromate reaction when present in sufficient excess, whilst in the ammonium vanadate test they give a fugitive green coloration (stable in the case of salicylic acid). The characteristic strychnine reactions are modified or retarded by an excess of citric acid, pyperonylic acid, quinine, glucose and mannitol. On the other hand, the colorations given by the anilides (acetanilide, benzanilide, etc.) with potassium bichromate may be readily distinguished from the violet coloration given by strychnine, and do not mask the latter. Among the numerous therapeutic agents that do not mask the strychnine reactions are caffeine, salophen, theobromine, saccharin, saligenin, sulphonal, trional, tetronal, veronal, and antithermin. For the separation of strychnine from the various organic compounds the author makes use of the fact that it is nearly insoluble in cold water, absolute alcohol, and anhydrous ether, scarcely soluble in boiling water (1 : 2,500), sparingly soluble in 90 per cent. alcohol (1 : 107), in benzene (1 : 165), in carbon tetrachloride (1 : 155), in amyl alcohol (1 : 180), and in glycerol, petroleum spirit, and carbon bisulphide; whilst, on the other hand, it is readily soluble in chloroform (1 : 7). A good method is to extract the mixture with chloroform, to evaporate the extract, and to treat the dry residue with anhydrous ether, which will remove the bulk of the substances that would interfere with the test for strychnine. C. A. M.

Estimation of Sulphur by the Method of Carius. G. Anelli. (*Gazz. Chim. Ital.*, 1911, **41**, 334-341.)—Estimation of sulphur by the method of Carius, as used by Peratoner (*Chem. Zentralbl.*, 1909, **1**, 479)—viz., heating the sample at 180° to 200° C., for four to eight hours with fuming nitric acid in a sealed tube—is shown to give results invariably too high. Thus, estimations of the sulphur in samples of pure sulphur and mixtures of sulphur and bitumen gave results ranging from 101 to 106 per cent. of the theoretical amount. This was found to be due to the action of the sulphuric acid, formed in the reaction, upon the silica in the glass, and the subsequent precipitation of the silicic acid as barium silicate. This may be obviated by placing barium nitrate in the tube, so that the sulphuric acid is precipitated as soon as it is formed. C. A. M.

Estimation of Theobromine and Caffeine in the Presence of Each Other.

C. Monthulé. (*Ann. Chim. anal. appl.*, 1911, **16**, 137-138.)—When a mixture of theobromine and caffeine is extracted with petroleum spirit, it is often difficult to dissolve the whole of the caffeine, especially when alkalis are present. This difficulty may be obviated by the use of the following modification of Kunze's method, in which the ammoniacal solution of the alkaloids is neutralised with acetic acid, so as to obtain a gelatinous precipitate containing the whole of the theobromine, whilst the caffeine remains in solution. The weighed quantity (0·2 grm.) of the mixed alkaloids is dissolved in a little ammonia solution in a 100 c.c. flask, and the solution treated with 20 c.c. of $\frac{N}{10}$ silver nitrate solution, any precipitate that forms being dissolved by the addition of more ammonia. The liquid is diluted, neutralised with acetic acid (with phenolphthaleïn as indicator), made up to 100 c.c., and filtered. Half of the filtrate is titrated with $\frac{N}{10}$ thiocyanate solution by Volhard's method to obtain the amount of uncombined silver. The percentage weight of theobromine is calculated by means of the formula $(10 - n) \times 16·6$, where n represents the number of c.c. of thiocyanate solution. Obviously the method is not applicable in the presence of chlorides, bromides, iodides, etc. The amount of caffeine may be found by evaporating another portion of the filtrate upon the water-bath in the presence of sodium chloride, and extracting the residue with chloroform. C. A. M.

Use of Temperature Corrections in the Polarisation of Raw Sugars and Other Products upon Quartz Wedge Saccharimeters.

C. A. Browne. (*Seventh Inter. Cong. App. Chem.*, 1909, Section V., 115-141.)—As the result of a great number of determinations, and from theoretical consideration of the effect of temperature on the specific rotation of sucrose and lævulose, and on the polarisation of mixtures of sucrose and sugar cane molasses, the author concludes that no temperature correction can be applied which will be of satisfactorily general significance for all types of commercial sugar products. Any temperature correction which is embodied in the adjustment of the saccharimeter—*e.g.*, to make an instrument adjusted at 20° C. give the same results as one adjusted at 30° C., when both are used at their specified temperatures—can only be valid for solutions of approximately pure sucrose. No such correction can take into account the influence of temperature on the specific rotation of the impurities, varied in their nature and amount, present in all types of raw products. Even if a correction table were drawn up for a certain type of raw sugar, its indications would be false for other sugars equally impure but varying from the average type. If temperature corrections are to be legitimately applied, it will be necessary to correct for changes in the specific rotations of all the ingredients present in the sample. Consequently for accurately comparative work, all sugar laboratories should be provided with means for warming or cooling the solutions to be examined to a standard uniform temperature throughout the year. J. F. B.

Estimation of Ash in Sugar and Syrups by the Determination of Electrical Conductivity. H. Main. (*Seventh Inter. Cong. App. Chem.*, 1909, Section V., 66-68.) — Considering sugar as a non-electrolyte, the electrical conductivity of its solutions may be regarded as a quantitative function of the salts present. The author has based a method for the estimation of ash in sugar on measurements of the resistances of solutions by Kohlrausch's method. The cell in which the resistances are measured should be fitted up on the "continuous" principle, like the continuous polarisation tube. Resistances are measured with the aid of a resistance-box, a "bridge" and a telephone. Although theoretically the sugar has no conductivity, practically the concentration of the sugar solution has an influence which may be eliminated by examining solutions of approximately the same density. The method is standardised against the incineration method for various types of sugar products, on the assumption that the relations between the constituents of the ash are the same for samples of the same type. For sugars containing 2 to 5 per cent. of ash, 5 grms. are dissolved to 100 c.c.; in the case of molasses the syrup is diluted to a density of 33° B. and 2 grms. are dissolved to 100 c.c. The method is stated to give satisfactory results when the percentage of ash is calculated from the standardisation-factor for the corresponding type of sugar product. J. F. B.

Estimation of Reducing Sugars. S. R. Benedict. (*J. Biol. Chem.*, 1911, **9**, 57-59.)—The following solution is recommended for use in place of Fehling's solution in the volumetric estimation of reducing sugars: Copper sulphate, 18; sodium carbonate, 200; potassium thiocyanate, 125 grms.; 5 per cent. potassium ferrocyanide solution, 5 c.c.; and water up to 1 litre. Twenty-five c.c. of this solution are reduced by 0·05 grm. of dextrose or by 0·053 grm. of lævulose. The cuprous oxide is not precipitated owing to the presence of the ferrocyanide, and the disappearance of the blue colour indicates the end-point of the titration.

Methods for Testing Coal Tar and Refined Tars, Oils, and Pitches derived therefrom. S. R. Church. (*J. Ind. Eng. Chem.*, 1911, **3**, 227-233.)—Methods are described in detail, and all the apparatus is illustrated by drawings, many of which are drawn to scale. The methods cover the determination of the sp. gr. and viscosity of tar as well as the estimation in it of moisture, free carbon, fixed carbon, and ash. An additional sulphonation test for creosote oils is also described.

G. C. J.

Estimation of Tarry Substances in Dark Mineral Cylinder Oils. D. Holde and G. Meyerheim. (*Chem. Zeit.*, 1911, **35**, 369-370.) In estimating the tarry substances in dark mineral cylinder oils by treating the sample with a mixture of alcohol and ether, and subsequently removing the paraffin wax from the insoluble residue by means of alcohol, erroneous results are liable to be obtained unless the washing is continued for a very long period. This may be obviated by extracting the mixture of tarry substances and paraffin wax in a cartridge suspended in a flask beneath a reflux condenser. About 5 grms. of the well-mixed sample are dissolved in about 25 times the volume of ether, and the solution is treated, drop by drop with continual shaking, with 12·5 times the volume (calculated upon an oil of sp. gr. 0·91) of 96 per cent. alcohol. After a thorough shaking, and having stood for five hours in the closed flask at 15° C., the liquid is filtered, the insoluble residue washed with alcohol-ether (1 : 2) until the washings are free from oily particles, and dissolved in benzene, the solution evaporated, and the residue of tarry matters and paraffin wax dried for ten minutes at 105° C. and weighed. It is then dissolved in 10 c.c. of ether, the solution mixed with about 2 grms. of dry coarse pure bone charcoal (previously extracted with alcohol) and about 10 to 15 grms. of coarse ignited sand, and the ether evaporated. The mass is now introduced into an extraction cartridge, and extracted with boiling absolute alcohol (as described above) until paraffin wax is no longer extracted. The alcoholic extract is evaporated, the residue dried for ten minutes at 105° C., and the weight of the paraffin wax deducted from the combined weight of the paraffin wax and tarry matters previously obtained. C. A. M.

New Method of Estimating Tar and Tar Contituents in Gas. W. Feld. (*J. Gasbeleucht.*, 1911, **54**, 33-35; through *Chem. Zentralbl.*, 1911, **1**, 693.)—The gas, in which it is desired to estimate the amount of tarry substances which will be deposited from the gas at any given temperature, is passed through a series of U-tubes packed with cotton-wool, and previously dried and weighed. The tubes are supported in a water-bath at the required temperature during this part of the operation, and the tarry matters collected on the cotton-wool are then dried by passing the same gas through the tubes after this gas has been dried. As it is already saturated with tarry matters, the gas will now only remove water from the tubes.

W. P. S.

INORGANIC ANALYSIS.

Precipitation of Aluminium Hydroxide in Granular Form. W. E. Taylor. (*Chem. News*, 1911, **103**, 169.)—A recently-described method for obtaining a granular precipitate of alumina by the use of a potassium iodide and iodate mixture, is mentioned by the author, who, however, finds that the simplest and most satisfactory mode of operating is to precipitate the aluminium hydroxide by means of ammonia at a temperature of 66° C. The solution may subsequently be boiled before filtration, the precipitate being granular and filtering rapidly. A. R. T.

Volumetric Method for Antimony in Alloys. G. S. Jamieson. (*J. Ind. Eng. Chem.*, 1911, **3**, 250-251.)—The drillings are heated with 10 c.c. concentrated sulphuric acid until the alloy is decomposed. After the lead sulphate has become white, the solution is boiled for two minutes, and then cooled and diluted with 15 c.c. of water. When cool again, 15 c.c. of hydrochloric acid (1 : 1) are added, the mixture shaken well, and the lead sulphate filtered off on a Gooch crucible and washed with small quantities of hydrochloric acid (1 : 1). The filtrate is transferred to a 250 c.c.

stoppered bottle, and 5 c.c. chloroform, 15 c.c. of concentrated hydrochloric acid, and 5 c.c. of iodine monochloride solution, are added. The mixture is shaken, allowed to stand five minutes, and the liberated iodine then titrated with standard potassium iodate solution (*cf.* Andrews, ANALYST, 1903, **28**, 306) until the chloroform is just decolorised after thorough shaking, which should be repeated in about a minute to make sure of getting the true end-point. The iodine monochloride solution is prepared by dissolving 10 grms. potassium iodide and 6·44 grms. potassium iodate in 75 c.c. of water, adding 75 c.c. of concentrated hydrochloric acid and a drop of chloroform, and adjusting exactly to faint iodine colour by shaking and adding dilute potassium iodide or iodate, as the case may require. Neither copper nor iron interferes. The final titration must take place in acid of at least 15 per cent. strength, as shown by Andrews (*loc. cit.*). G. C. J.

Attempts to Prepare a Solid Hydride of Antimony. H. Reckleben and J. Scheiber. (*Zeitsch. anorg. Chem.*, 1911, **70**, 275-281.)—Attempts were made to prepare a solid hydride of antimony in the manner in which its preparation has been described by Ruhland (1815) and Marchand (1845), and by methods analogous to those which give rise to the solid hydride of arsenic. The products were analysed by a method similar to that employed by the same authors in the investigation of the solid hydride of arsenic (see next abstract), but using, instead of silver nitrate, iodine solution, which could not be used in the arsenic research because it did not dissolve the arsenic precipitates quickly enough. The products were in every case found to consist of nearly pure antimony, and no evidence was obtained which would justify the assumption that any solid hydride of antimony, analogous to that of arsenic, exists.

The so-called "explosive" antimony (a solid solution of antimony chloride in a metastable modification of metallic antimony) was investigated, as it has been suspected that this substance might contain a solid hydride, but the result was again negative. G. C. J.

Detection of the Solid Hydride of Arsenic. H. Reckleben and J. Scheiber. (*Zeitsch. anorg. Chem.*, 1911, **70**, 255-274.)—Though an alleged solid hydride of arsenic was described nearly 100 years ago, and has been the object of many researches since, its formula and even its existence have been a matter of doubt until the present research was undertaken. The formula As_2H_2 is that most often given in modern textbooks, but results have been published which seemed to point to the formula As_2H or AsH_2. The arsenic content of any of these compounds is so near 100 per cent. that the experimental error of an arsenic determination precludes it as a means of determining the formula. Oxidation of the compound followed by weighing the water formed is unsatisfactory, as the compound is usually the product of a reaction in aqueous solution, and all authors agree that it is difficult to obtain it dry without risk of decomposition. Liberation of the hydrogen followed by its measurement is also unconvincing, as the product is never free from elementary arsenic, which may conceivably reduce some water adhering to an imperfectly dried product. The present authors have made use of the reducing power of the compound compared with its arsenic content. The oxidising solution used was an ammoniacal solution of silver nitrate. One atom of arsenic reduces 5 atoms of silver, one molecule of AsH should reduce 6 atoms of silver, whilst one of AsH_2 should reduce 7 atoms of silver. In no case did the silver-arsenic ratio exceed 6 : 1, so that the formula AsH_2 seems excluded. On the other hand, in many preparations the ratio was notably less than 6 : 1, but never so low as 5·5 : 1, the ratio which would correspond to the formula As_2H. There was evidence that the material examined always contained some elementary arsenic, and the authors conclude that they are justified in assuming that their preparations were mixtures of AsH (or As_2H_2) with elementary arsenic, and in calculating the proportions of this binary mixture from its reducing power.

It is suggested that the method may be useful in the analysis of other mixtures of two, but not more than two, reducing substances of different reducing power.

G. C. J.

Proportion of Lead Arsenate in the Products of Vines treated with Arsenical Washes. L. Moreau and E. Vinet. (*Comptes rend.*, 1911, **152**, 1057-1060.)—In the case of vines treated before the flowering period with lead arsenate washes, the quantity of lead arsenate adhering to the grapes at the vintage period is very small, and a large proportion thereof is eliminated during the pressing and fermentation. Hence, only insignificant traces of lead and arsenic will be present in the wines, comparable with those found in wines from vines that have never been treated with arsenical washes. If, however, the vines are treated at a later period, a considerable proportion of lead arsenate will be taken up by the must, and will be found in the finished wine. Thus, in an experiment made by the authors, the vine was treated with the arsenic in August, and the following amounts of lead arsenate were found in the wine and various residues:

	Grapes.	Fresh Marcs.	Sediment after Fermentation.	Lees of First Racking.	Lees of Second Racking.	Wine from Second Racking.
Weight corresponding to 1 hl. of wine, kilos. ...	153·7	37·3	6·2	1·7	1·5	1 hl.
Lead arsenate, grms. ...	7·165	6·117	0·582	0·115	0·015	0·414

C. A. M.

Sulphite Method for Separating and Identifying Calcium and Strontium. J. I. D. Hinds. (*J. Amer. Chem. Soc.*, 1911, **33**, 510-514.)—When a saturated solution of sodium sulphite is added to a solution of strontium chloride made strongly acid with acetic acid and the mixture boiled, a distinct precipitate results if the strontium concentration exceeds 0·0015 normal. Calcium sulphite is not very soluble in water, but in solutions made strongly acid with acetic acid it is not precipitated until the concentration approximates 1·3 normal. Strontium sulphite is even less soluble in neutral or alkaline than in acetic acid solution, but the difference in solubility of strontium and calcium sulphites is most marked in solutions made strongly acid with acetic acid. G. C. J.

Estimation of Copper by Means of Hypophosphorous Acid. J. Hanůs and A. Soukup. (*Zeitsch. anorg. Chem.*, 1911, **70**, 282-293.)—The apparently accurate results obtained when the Mawrow-Muthmann method (*Zeitsch. anorg. Chem.*, 1896, **11**, 268) is applied under favourable conditions depend on an approximate compensation of errors which are individually large and which may be very far from being mutually compensating. The positive error is mainly due to the oxidation of the separated copper, the negative error to incomplete precipitation, and sometimes to re-solution of the separated and oxidised copper. The precipitation is never quantitative, and the quantity of copper remaining in solution depends on the concentration of hydrion, on the time of contact of the copper with air, and of the oxidised copper with the acid solution. The concentration of hydrion is conditioned by the reaction itself, the excess of hypophosphorous acid and its purity, and finally, when sodium or magnesium hypophosphite is used, by the amount of acid used to accelerate the reaction. Only by repeated, cautious neutralisation can satisfactory results be obtained. Prolonged boiling must also be avoided, otherwise some of the hypophosphorous acid will be oxidised. The use of sodium acetate or tartrate to reduce the concentration of hydrion is without measurable influence on the results. The method cannot be made exact, and is unsuited to the separation of copper from cadmium, zinc, and other metals. G. C. J.

Dangers connected with the Storage and Transport of Ferrosilicon. C. Göpner. (*Chemische Industrie*, 1911, **34**, 31-38 and 64-72.)—An abstract of Copeman's report to the Local Government Board. It has been stated by various observers that, when acted on by water or moist air, ferrosilicon may give rise to hydrogen, acetylene, and the hydrides of phosphorus and arsenic, as well as that of silicon. Hake, who analysed the gases from some seventy samples collected by Copeman, found no trace of acetylene or silicon hydride. Hydrogen, as non-poisonous, was not looked for. The hydrides of phosphorus and arsenic were estimated in every case in which a qualitative examination showed their presence. Of the sixteen samples with 35 per cent. or less silicon, only two gave rise to a detectable trace of phosphoretted hydrogen, and none of these samples yielded any arseniuretted hydrogen. Most of the samples with 42 per cent. or more silicon gave rise to quantities of phosphoretted hydrogen which could be estimated gravimetrically, and many of them to notable amounts of arseniuretted hydrogen. Since the ratio of the volume of the former gas to that of the latter was invariably of the order of 10 : 1, it may be assumed that the chief danger of ferrosilicon lies in the risk of evolution of phosphoretted hydrogen. It is well known that the explosions which have occasionally accompanied the transport of ferrosilicon have been in every case caused by material of medium (40 to 60 per cent.) silicon content, and it has been said that material with more or less silicon than 60 or 40 per cent. respectively is safe as regards evolution of poisonous gases. Hake's results tend to confirm the view that low-grade ferrosilicons are not a source of danger, but the same cannot be said of the very high-grade ones. It is, however, noteworthy that the largest quantities of poisonous gases were obtained from samples containing 42 to 52 per cent. of silicon. Hake's analytical methods are described. Phosphoretted hydrogen was estimated either by passing it through a 0·5 per cent. solution of gold chloride and weighing the reduced gold, or by passing it through a measured quantity of $\frac{N}{10}$ silver nitrate, and then proceeding in one of the following ways: either the solution was filtered from silver phosphide, and the excess of silver in the filtrate determined by Volhard's method, or the silver phosphide was oxidised by means of nitric acid, the nitric acid removed by evaporation, silver precipitated by means of hydrogen sulphide, and the phosphoric acid in the filtrate finally obtained as magnesium pyrophosphate. The three methods give concordant results, and the volumetric method alone was employed in many experiments. Arseniuretted hydrogen was estimated by leading the gases through a solution of silver nitrate, filtering from silver phosphide, and adding the filtrate to a Marsh apparatus. The paper ends with a literal translation of Copeman's recommendations, which include the suggestion that ferrosilicon should not be packed or despatched from the works of its origin until a month after breaking to the customary size, and the further suggestion that sufficient hay or other packing material be used to prevent any movement of the contents of the barrels. G. C. J.

Estimation of Fluorine. G. Starck. (*Zeitsch. anorg. Chem.*, 1911, **70**, 173-177.)—The method described depends on the comparative insolubility of the compound PbFCl in water, and its still more marked insolubility in solutions of lead chloride. It is more soluble in dilute acids than in water. A solution of a fluoride is therefore first neutralised, and then mixed with a large volume (200 c.c.) of a nearly saturated solution of lead chloride, and the mixture allowed to stand overnight. The precipitate, which weighs fourteen times as much as the fluorine contained in it, is filtered off on a Gooch crucible, well washed with lead chloride solution, and finally washed three or four times with a small quantity of water. It is dried at 140° to 150° C. for two hours and weighed. At 18° C., 100 c.c. of water dissolve 32 mgrms.; 100 c.c. of $\frac{N}{20}$ lead chloride solution dissolve 0·2 mgrm. Quantities like 0·03 grm. of fluorine, corresponding to about 0·4 grm. of the lead compound, can be estimated with an error not exceeding ± 1 per cent. G. C. J.

Quantitative Analysis of Gold with the Use of Ether. F. Mylius. (*Zeitsch. anorg. Chem.*, 1911, **70**, 203-231.)—The author proposes to take advantage of the solubility of gold chloride in ether as a means of separating gold from other metals. The aqueous solution of gold should be strongly acid with hydrochloric acid, as the gold then exists as $HAuCl_4$, which is more soluble in ether and less soluble in water than gold chloride. The presence of a little nitric acid is also an advantage, so that a solution of the metal in aqua regia, after dilution until the concentration of hydrogen chloride is about 10 per cent., may be submitted to extraction at once without the necessity for eliminating any of the acid. When the gold needs only to be estimated accurately to within ± 0·1 per cent., four or five extractions with ether complete the analysis. But it is shown that the method may be employed to increase the accuracy as well as to diminish the tediousness of the most accurate class of work required at a mint. In such cases the ether process is made use of to separate the bulk of the gold from all the other metals. These and traces of gold are then recovered from the acid-aqueous solution by the usual methods. Special apparatus for conducting the ether extractions is described, and detailed directions are given for making a complete analysis of gold coinage. Test analyses of the gold coins of eight countries are given, and in every case, save two, the percentage of gold found was 90·000 per cent. The exceptions were English sovereigns and some old Russian coins which complied exactly with the English standard of 91·667 per cent.

Incidentally it is shown that the ether method affords the most ready means of preparing normal gold of the highest grade of purity. G. C. J.

The Influence of Pertitanic Acid on the Estimation of Iron in Minerals. E. Knecht and E. Hibbert. (*J. Soc. Chem. Ind.*, 1911, **30**, 396-397.)—The direct volumetric estimation of ferric iron in presence of titanic salts presents no difficulty if any of the usual reduction methods are employed, but if a fusion, in the case of refractory minerals, has been made, erroneous results may be obtained. Thus, minerals like rutile and ilmenite are only slowly attacked by fusion mixture, though they are readily and completely resolved by fusing for ten minutes with potassium hydroxide in a nickel crucible; if then the melt be taken up with water and the solution strongly acidified with hydrochloric or sulphuric acid, both the iron and titanium pass into solution, and an orange coloration of the liquid is produced, due to the presence of pertitanic acid (TiO_3). This pertitanic acid, if not first destroyed, causes high results in the titration of the iron. Larger amounts of pertitanic acid are produced in the case of nickel vessels than when silver vessels are used. The authors propose to overcome this interference with the method by running into the sulphuric acid solution permanganate solution till a faint permanent pink coloration is obtained. Pertitanic acid then acts like hydrogen peroxide, being completely reduced to titanic oxide, with evolution of oxygen. The titration of the iron in the solution, in presence of potassium sulphocyanide, by means of titanous chloride is then proceeded with as usual. The amount of pertitanic acid formed in these fusions is very considerable, nearly 15 per cent. of pertitanic acid, calculated on the titanic oxide present, being produced in one case. Its formation is independent of the presence of iron. A. R. T.

Method for Testing Galvanised Iron to Replace the Preece Test. W. A. Patrick and W. H. Walker. (*J. Ind. Eng. Chem.*, 1911, **3**, 239-242.)—The so-called Preece test consists in placing the test piece of galvanised iron in a solution of copper sulphate under standard conditions, and observing the number of one-minute immersions which can be made before copper in a bright adherent form will plate out on the article. It assumes that the galvanised coating is homogeneous, and that no bright copper will be seen until the iron base is uncovered. Neither of these assumptions is justified. Galvanised coatings usually include a well-defined layer of iron-zinc alloy between the iron base and the outer layer of pure zinc. The rate of solution of this alloy is very different from that of zinc, and under certain conditions bright copper may be deposited on the alloy long before the iron base is exposed. Moreover, the ratio of the thickness of the alloy to that of the whole coating may vary within wide limits. In some wet galvanised (electro-deposited) sheet the alloy may be represented by an insignificant film. In other sheets made by the same process, and in all "Sherardised" plate, the alloy forms a comparatively thick and well-defined layer. In hot-dipped sheet it forms a well-defined but usually thin layer.

The following method is recommended in substitution for the Preece test: The test piece is immersed for three minutes in a solution of basic lead acetate. The adherent lead is removed by a stiff brush, avoiding any burnishing action, and the immersion and brushing repeated until a bright surface of iron is exposed. Four immersions of three minutes each usually suffice. The coating is determined either from the loss in weight of the test piece or from the weight of reduced lead, which is either weighed as such or dissolved and estimated by a volumetric method. Since as much as 10 per cent. of the coating may be iron alloyed with zinc, the iron passing into solution as acetate should be estimated. Samples of weathered galvanised goods must be cleaned in dilute hydrochloric acid before testing. The basic lead acetate solution is made by shaking together 400 grms. lead acetate, 4 grms. litharge, and 1,000 c.c. water. The clear solution is decanted from any insoluble residue and diluted to a sp. gr. of 1,275. Such a solution dissolves zinc-iron alloys, but does not attack iron. G. C. J.

Estimation of Manganese by the Bismuthate Method. P. H. M. P. Brinton. (*J. Ind. Eng. Chem.*, 1911, **3**, 237-239.)—The conclusion of other workers is confirmed that, for the estimation of small amounts of manganese, the bismuthate method is the most accurate known. When the permanganate is standardised against oxalate or iron, manganese is sensibly underestimated. A gravimetrically standardised solution of manganese sulphate is the correct primary standard; but, as this method is less convenient than the sodium oxalate method, it is suggested that pure Sörensen sodium oxalate be used, and that the calculated manganese titer be corrected in accordance with the results published in the present paper. The theoretical value of the ratio 5 $Na_2C_2O_4$: 2 Mn is 1 : 0·16024. It is shown that the empirical factor 0·1656 is to be preferred in calculating the manganese titer of the permanganate. G. C. J.

Estimation of Soluble Mercuric Salts at Great Dilutions. H. R. Procter and R. A. Seymour-Jones. (*J. Soc. Chem. Ind.*, 1911, **30**, 404.)—The method was devised for the determination of quantities of mercuric chloride as small as 1 part in 100,000 of water, and takes advantage of the fact that certain organic acids (such as formic and citric) prevent the precipitation of mercuric sulphide, this compound remaining in the coloured colloidal form in solution on treatment with hydrogen sulphide. The coloration produced is perfectly definite for prescribed concentrations of the mercuric chloride and acid if the liquid has been saturated with hydrogen sulphide, and is directly proportional to the concentration of the original mercuric chloride solution. Thus, when certain thicknesses of liquid are examined and compared with a standard, using a Schmidt and Haensch "dipping colorimeter" (*J. Soc. Chem. Ind.*, 1910, **29**, 663), a solution of half the strength will require to be twice the thickness to produce the same colour. A standard solution containing 1 part of mercuric chloride by weight in 10,000 of water, and 1 per cent. by weight of formic acid, is saturated with hydrogen sulphide gas and placed in a standard 1 c.c. cell on the upper platform of the colorimeter. Against this standard colour all solutions of lower concentration may be matched. The mercuric solution of unknown strength is made into a 1 per cent. formic acid solution, similarly treated with hydrogen sulphide and run into the dipping cylinder of the colorimeter. The dipping stage is then adjusted till the field of view in the telescope is of a uniform colour, when the depth of solution is read off and its strength calculated, the depth of the standard liquid being 10 mm. It is possible to distinguish by this method between 76 and 77 parts of mercuric salt per 100,000. Small quantities of arsenic and lead may be similarly determined, suitable standards being prepared. Lovibond standard glasses may be used instead of a liquid standard, the glasses being matched with the prepared standard in the usual way. A. R. T.

Quantitative Analysis of Mixtures by Differences in the Specific Gravities of their Components. H. Friedenthal. (*Ber.*, 1911, **44**, 904-909.)—In many cases, especially in physiological analysis—*e.g.*, of blood, milk, wine, gall, etc.—a quantitative separation of the constituents may be effected by evaporating the substances to dryness, reducing to a fine powder, and separating the components by the differences between their specific gravities. With mixtures of bromoform, sp. gr. 2·90, and xylene, sp. gr. 0·70, a suspension liquid of any density desired between these limits is readily obtained. In simple cases, separation is effected in an ordinary separating funnel; in other cases a centrifugal tube having a stopcock of full bore in the middle may have to be employed. The stopcock has no projecting handle liable to fracture, but is operated by a key. As a simple example, the author quotes the quantitative separation of small quantities of potassium chloride (sp. gr. 1·98) from sodium chloride (sp. gr. 2·10) by means of a suspension liquid of sp. gr. 2·04. The author is engaged in the study of a more complicated phase of the same question: the separation of dissolved and colloidal bodies by the application of extremely high centrifugal forces. Special machines have been constructed making 10,000 revolutions per minute, and plans for a machine giving 100,000 revolutions are under consideration. The machine has to be very accurately balanced and well protected. With these fast machines, the casein can be separated from the lactalbumin of milk; the fat can be separated into liquid and solid fractions. Combination with iodine or bromine has the effect of greatly accentuating the differences in the specific gravities of various organic substances. For the separation of solid substances, the material should first be freed from fat, the residue should be dried *in vacuo*, and then powdered in an electrically-operated agate grinding machine; the condition of the grinding may be controlled without appreciable loss by microscopic examination J. F. B.

Rapid Determination of Nickel and Cobalt by Means of the Gauze Cathode and Stationary Anode. R. C. Benner and W. H. Ross. (*J. Amer. Chem. Soc.*, 1911, **33**, 493-504.)—It is shown that the rapid estimation of nickel and of cobalt by means of the gauze electrode and stationary anode is perfectly satisfactory with most of the electrolytes in common use. The rate at which they are precipitated is practically the same for all the electrolytes which can be used in ammoniacal solution, and is little affected by the amount of ammonia added. In acid solution the rate of precipitation is not quite as great as in ammoniacal solution, and decreases slightly as the amount of acid is increased. Although the rate of precipitation of these elements on the gauze electrode is not quite equal to the rate at which they are precipitated by means of the same current when the electrolyte is agitated by mechanical means, it is rapid enough for practical purposes. In addition to its simplicity, the apparatus has the advantage that it can, when desired, be used without any alteration with the small currents, which alone were used formerly and which it is still sometimes convenient to employ. G. C. J.

New Method of Estimating Nitrites. E. Rupp and F. Lehmann. (*Arch. Pharm.*, 1911, **249**, 214-217.)—The method depends upon the oxidation of the nitrous acid to nitric acid by means of bromine, the excess of which is estimated iodometrically. In practice it is better to use nascent bromine liberated from an acid solution of potassium bromate and bromide. In estimating—*e.g.*, sodium nitrite—2·5 grms. of the sample are dissolved in 500 c.c. of water, and 10 c.c. of the solution mixed in a stoppered flask with 50 c.c. each of $\frac{N}{100}$ potassium bromate solution and potassium bromide solution (6 grms. of the dry salt per litre), and 10 c.c. of dilute sulphuric acid. The flask is then well shaken and allowed to stand for thirty minutes in the dark, after which 0·5 grm. of potassium iodide is introduced, the flask again shaken, and after two minutes the liberated iodine titrated with $\frac{N}{10}$ sodium thiosulphate solution. The number of c.c. required is deducted from 30 c.c. (the quantity corresponding to the bromate solution taken), and the difference multiplied by the factor 0·00345 gives the quantity of sodium nitrite present.

C. A. M.

Colorimetric Method of Estimating Phosphoric Acid. N. Passerini. (*Gazz. Chim. Ital.*, 1911, **41**, 182-186.)—A colorimetric method of estimating phosphoric acid is based upon the coloration given by molybdic acid with gallic acid, the colour being yellow, orange, or red, according to the amount of gallic acid. A similar coloration is obtained with gallotannic acid, but that substance is less suitable for the purpose, since it is usually more or less coloured. The phosphoric acid is dissolved from the rock, etc., by means of nitric acid, and precipitated from an aliquot portion of the solution with the usual molybdic reagent. After twenty-four hours, the precipitate is collected, washed with water containing 1 per cent. of nitric acid until the filtrate no longer gives a yellow coloration with gallic acid, and dissolved in the smallest possible quantity of dilute (1 : 4) ammonia solution. The liquid is boiled until it no longer smells of ammonia, and is then cooled and made up to 100 c.c. For the colorimetric estimation 15 c.c. of water and 2 c.c. of a freshly-prepared saturated solution of gallic acid are placed in each of two glass cylinders of equal diameter. Into one of these tubes is introduced 1, 1·5, or at most 2 c.c., of the standard solution of phosphoric acid, whilst the solution under examination is run drop by drop from a burette, with continual shaking, until the colours of the two liquids match. The standard solution (1 c.c. = 0·01 mgrm. of P_2O_5) may be prepared from any phosphate of known composition. Thus, 1·9153 grms. of pure dicalcium phosphate are dissolved in 1 litre of water acidified with 1 per cent. of nitric acid, and 10 c.c. of the solution (= 0·01 grm. of P_2O_5) are treated with 2 c.c. of a 75 per cent. solution of ammonium nitrate, and with 25 c.c. of nitro-molybdic reagent, and the precipitate allowed to stand for twenty-four hours, washed with 1 per cent. nitric acid, and dissolved in ammonia solution. The liquid is filtered, and the filtrate and washings made up to a litre. This slightly ammoniacal solution will keep unchanged for a long time. When required for an analysis, a portion of it is boiled to remove the bulk of the free ammonia, and made up to its original volume. The tabulated results quoted show that the method gives results in close agreement with those obtained by the gravimetric method, whilst, in the case of materials containing only very small quantities of phosphoric acid, it is somewhat more accurate.

C. A. M.

Volumetric Estimation of Phosphoric Acid Soluble in 2 Per Cent. Citric Acid Solution. L. Wuyts. (*Ann. Chim. anal. appl.*, 1911, **16**, 134-137.)—The following method is recommended as giving results in close agreement with those obtained by the nitro-molybdate method: Ten c.c. of the solution (5 grms. of the sample to 500 c.c. of 2 per cent. citric acid solution) are treated with a few drops of hydrochloric acid and evaporated to dryness, and the residue taken up with 3 to 4 drops of nitric acid and a few c.c. of water. The solution is treated with 10 c.c. of nitric acid (sp. gr. 1·40) and 15 c.c. of a saturated solution of ammonium nitrate and heated to the boiling-point. It is next allowed to stand for two or three minutes and treated with 25 c.c. of ammonium molybdate solution (90 grms. per litre), and the resulting precipitate stirred up, allowed to settle again for about twelve minutes, collected, and washed free from acid with cold water. It is then dissolved in standard potassium hydroxide solution (326·5 c.c. of $\frac{N}{1}$ solution diluted to a litre; 1 c.c. = 1 mgrm. of phosphoric acid), an excess of 1 or 2 c.c. being added, and the solution treated with 0·5 c.c. of phenolphthaleïn solution (1 grm. in 100 c.c. of 60 per cent. alcohol) and about 50 c.c. of water, and titrated with standard sulphuric acid equivalent to the potassium hydroxide solution. The number of c.c. used, less the quantity required to neutralise the excess of alkali, gives directly the percentage of phosphoric acid.

C. A. M.

Estimation of Silver by Electrolysis of an Ammoniacal Solution of its Oxalate. F. A. Gooch and J. P. Feiser. (*Zeitsch. anorg. Chem.*, 1911, **70**, 294-296.)—Silver may be deposited from an ammoniacal solution of its oxalate in a state of purity, and suitable for its quantitative estimation. Ammonium nitrate or chloride may be present. The cathode surface should preferably be relatively large and the current density low, the exact value necessarily depending on the form of cathode, of which several were tried, and in every case rotated about 400 to 500 times a minute. Half a grm. of silver may be deposited in half an hour. There being no non-volatile substance in the exhausted solution, the results are never high, but may be as much as 0·7 mgrm. low. G. C. J.

"Perhydrol" for the Estimation of Total Sulphur in Coal-Gas. M. Dickert. (*J. f. Gasbeleucht.*, 1911, **54**, 182-183; *Chem. Zentralbl.*, 1911, **1**, 1154.)—Seventy-five c.c. of sodium hydroxide solution of 30° B. are mixed with 10 c.c. of "perhydrol," the mass of crystals which form is broken up by stirring, and the reagent is placed in a Drehschmidt's gas-washing bottle, through which a current of the gas to be analysed is passed at a rate of up to 100 litres per hour. The contents of the bottle are subsequently washed into a beaker, acidified with hydrochloric acid, and boiled, and the sulphuric acid is either precipitated as barium sulphate or titrated according to Holliger's method. Thiophen appears to escape oxidation by this method, but the quantity of this substance in coal-gas is so small that its presence may be neglected. J. F. B.

Estimation of Sulphur in Coal-Gas and of Ammonium Sulphate. H. Blair. (*J. Soc. Chem. Ind.*, 1911, **30**, 397-398.)—The method depends upon the reaction between formaldehyde and an ammonium compound, hexamethylenetetramine being formed: $4NH_4Cl + 6CH_2O = N_4(CH_2)_6 + 4HCl + 6H_2O$. The acid may be titrated with alkali and phenolphthaleïn.

For Sulphur in Coal-Gas.—The liquid obtained in the "Referee's Test" is diluted to 500 c.c., and 50 c.c. boiled for two or three minutes to expel carbon dioxide. Ten c.c. of neutral formaldehyde solution (about 30 per cent.) are then added to the hot solution, and the liquid at once titrated with $\frac{N}{10}$ potassium hydroxide solution. One c.c. = 0·0247 grain of sulphur. The alkali should be standardised against a standard solution of ammonium sulphate, containing a little ammonium carbonate. Concordant results, slightly higher than are obtained by the barium sulphate method, are obtained. By this method a small quantity of gas (1 or 2 cubic feet) may be examined.

For Ammonium Sulphate.—The solution, always slightly acid, is neutralised by potassium hydroxide, boiled for a few minutes, and treated with 10 c.c. of neutral formaldehyde solution. After continuing the boiling for a few seconds, phenolphthaleïn is added, and the liquid titrated as before. One c.c. = ·0066 grm. $(NH_4)_2SO_4$. The results obtained agree with those given by the barium sulphate method. A. R. T.

Simple Method of Decomposing Refractory Tin Ores. H. Y. Loram. (*Proc. Chem. Soc.*, 1911, **27**, 60.)—The mineral (2 grms., or more if desired), crushed to a sixty-hole powder, is fused for about an hour in a covered silver crucible at a temperature well below its melting-point, with about six or seven times its weight of potassium hydroxide, to which is added potassium cyanide equal to the weight of tin ore taken.

The mass, on cooling, is extracted with water, when the whole will dissolve in dilute hydrochloric acid, and, after boiling to destroy any cyanide still remaining, the tin can be estimated by any of the usual methods.

Sensitiveness of the Colorimetric Estimation of Titanium. R. C. Wells. (*J. Amer. Chem. Soc.*, 1911, **33**, 504-506.)—The object of the experiments described in this paper was to establish the range of suitable concentrations for the colorimetric estimation of titanium. The solutions contained 3 per cent. of sulphuric acid and 2 per cent. of hydrogen peroxide. It is found that titanium may be estimated with equal accuracy over a rather wide range of concentrations—namely, from 1·5 to 20 mgrms. titanium dioxide per 100 c.c. Within these limits of concentration the determination may be made with an error of certainly less than 3 per cent. Most observers will find it easy, with a suitable colorimeter, to keep the mean error of their observations below 2 per cent. G. C. J.

Use of Methyl Orange in the Colorimetric Estimation of Titanium. A. Gautier. (*Rev. Generale Chim.*, 1911, **14**, 16-17; through *Chem. Zentralbl.*, 1911, **1**, 843.)—Methyl orange solutions may be used as comparison solutions in the colorimetric estimation of titanium; these solutions keep well, and their colour is not altered by exposure to the air. The concentrations of helianthin and titanium solutions of equal depth and tint are given in tabular form in the original paper.

W. P. S.

Indirect Method for the Estimation of Tantalum and Columbium. H. W. Foote and R. W. Langley. (*Amer. J. Sci.*, 1910, **30**, 393-400; through *Chem. Zentralbl.*, 1911, **1**, 1249.)—The method depends on the difference between the densities of the oxides of tantalum (8·716) and columbium (4·552). In the case of such a mineral as stibiotantalite, a quantity of the sample is treated with hydrofluoric acid, the solution is diluted with water, and the antimony and bismuth are removed by means of hydrogen sulphide. The solution containing the tantalum and columbium is then evaporated to dryness, the residue is heated with sulphuric acid to remove hydrofluoric acid, the hydroxides are precipitated from the diluted solution with ammonia, collected on a filter, washed, ignited over a blast-lamp, and weighed. The sp. gr. of the mixed ignited oxides is then determined, and the quantity of each present is calculated from the figure obtained (*cf.* ANALYST, 1911, 239).

W. P. S.

Estimation of Tungsten by Means of Stannous Chloride. B. Mdivani. (*Ann. Chim. anal. appl.*, 1911, **16**, 132-134.)—A method of estimating tungsten is based upon the fact that it may be precipitated by means of an excess of stannous chloride solution in the form of the oxide, W_2O_5. For example, the solution of 0·1 grm. of tungstic acid in 50 c.c. of dilute ammonia solution is treated with 20 c.c. of a freshly prepared solution of stannous chloride (50 grms.) in 200 c.c. of concentrated hydrochloric acid, and the mixture boiled for one or two minutes and allowed to stand. The precipitate is washed several times by decantation, and is finally collected, ignited, and weighed as tungsten trioxide. In the test experiments described the amounts of tungstic acid found ranged from 0·0995 to 0·1005 grm. The precipitate was quite free from tin, and it was proved that the presence of iron had no influence upon the results. C. A. M.

Estimation of the Salinity of Sea Water by Means of the Refractometer.

A. Berget. (*Comptes rend.*, 1911, **152**, 984-986.)—A series of determinations of the refractive indices, by means of Féry's refractometer, of solutions of sodium chloride have shown that they increase proportionately with the increase in the amount of salt. Thus the following results were obtained:

		0·010	0·015	0·020	0·025	0·030	0·035	0·040	0·045
Refractive index $[n]_{D\ 16^\circ C.}$	1·333804	1·33453	1·33536	1·33622	1·33712	1·33789	1·33875	1·33968	1·34050

In like manner a sample of sea water from Monaco gave the following results: Original water, 1·33929; diluted to half its salinity, 1·33576; and diluted to one-fourth, 1·33400 at 22·7° C. As in the case of the salt solutions, these results, when plotted graphically, formed a straight line. The method thus gives results quite as accurate as those calculated from the specific gravity. C. A. M.

Estimation of Silicon in Vanadium and Molybdenum and in their Iron Alloys.

W. Trautmann. (*Zeitsch. angew. Chem.*, 1911, **24**, 635-636.)—When the metals vanadium and molybdenum or their iron alloys are dissolved in the usual way, and the insoluble silica is collected on a filter, it is usually found that the silica has a green colour, owing to its being contaminated with traces of compounds of vanadium or molybdenum. The latter tend to form immediately after the acid solution has been decanted through the filter and the washing process is commenced. To remove these traces of impurities from the silica, it is recommended that, after the diluted acid solution of the metal has been decanted, the residue of silica should be washed twice with water, and then extracted with hot 10 per cent. hydrochloric acid until it is colourless. The silica is finally washed with water, dried, ignited, and weighed. In the case of molybdenum it is better to extract the silica with hot concentrated hydrochloric acid containing a little nitric acid. W. P. S.

Estimation of the Hardness of Water: the Wartha-Pfeiffer Method and its Modification.

J. M. Silber. (*Arch. f. Hyg.*, 1911, **73**, 171-182; *Chem. Zentralbl.*, 1911, **1**, 1081-1082.)—According to the original Wartha-Pfeiffer method (*Zeitsch. angew. Chem.*, 1902, **15**, 193), the temporary hardness in many cases is found to exceed the total hardness. The author describes the following modification, the solutions used being a 0·2 per cent. solution of methyl orange, $\frac{N}{10}$ hydrochloric acid, and a $\frac{N}{10}$ solution of a mixture of equal parts of sodium hydroxide and carbonate; 100 c.c. of the water are treated with 2 to 3 drops of the indicator, and titrated cold with $\frac{N}{10}$ hydrochloric acid to the red point.

The number of c.c. required is called *A*. The neutralised water is heated to boiling, treated with an excess of the $\frac{N}{10}$ alkali mixture, and further boiled for five minutes.

The liquid and precipitate are transferred quantitatively to a graduated stoppered cylinder, cooled, diluted to 200 c.c., and filtered; 100 c.c. of the filtrate are titrated back with $\frac{N}{10}$ acid; the number of c.c. is multiplied by 2 and subtracted from the number of c.c. of alkali added. The result, *B*, multiplied by 2·8, gives the total hardness (German degrees). Next, 100 c.c. of the original water are boiled for half an hour, keeping the volume constant, in order to decompose the bicarbonates. The filtrate is titrated with $\frac{N}{10}$ acid, and the number of c.c. of acid used, *C*, is subtracted from the original alkalinity of the water, *A*; and when the difference is multiplied by 2·8, the result is the temporary hardness. For the direct estimation of temporary hardness, 100 c.c. of the water are boiled in the usual way, filtered, and the precipitate is washed. The filter and precipitate are returned to the boiling-flask, treated with excess of $\frac{N}{10}$ hydrochloric acid, and the excess of acid is titrated back.

J. F. B.

APPARATUS, ETC.

Device for Accurately Reading a Burette. J. Milbauer. (*Chem. Zeit.*, 1911, **35**, 419.)—The principle of the Schnellbach burette may be applied to the reading of an ordinary burette. A sharp line, about 2 mm. in breadth, is drawn in red or blue ink upon a piece of cardboard, which is then cut with two slits, and placed over the burette, so as to form a background to the level of the liquid.

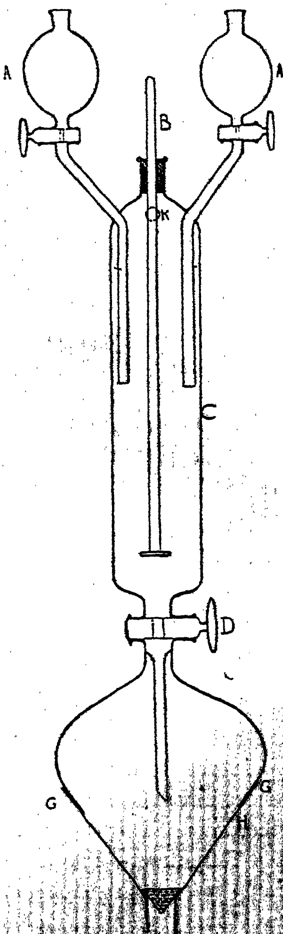
C. A. M.

Recording Thermo-Electric Calorimeter. C. Féry. (*Seventh Inter. Cong. App. Chem.*, 1909, Sect. X., 69.)—An apparatus is described which can be used continuously for checking the value of the gas produced in a gas works or producer plant. The gas is burnt at a constant rate in a special burner consuming from 5 to 10 litres per hour. The flame heats a recuperative thermopile, of which the cold junction is traversed by the air used for the combustion, and the hot junction is heated by the products of combustion. The pile contains fifteen junctions, enabling a P.D. of 0·2 volt to be maintained. The resistance being very low, the energy produced is sufficient to obtain a trace with a recording instrument similar to that in the Richard recording thermometers and barometers. For laboratory work this form is not so suitable as for everyday work on a gas of approximately uniform density, and the author has modified it by connecting the thermopile of the calorimeter to an electric meter which measures the energy developed by it, while the gas consumed is measured by a laboratory meter. As soon as a steady regime is set up, the ratio of the readings of the electric and gas meters gives the calorific power. It is advisable to regulate the consumption, so that the thermopile is in the same condition for different gases. With ordinary town gas a variation of 50 per cent. in the rate of consumption produced an error of proportionality of 3·5 per cent. With acetylene the variation was 1 per cent., and with gases of such different calorific powers as hydrogen and acetylene the proportionality is good, as can be seen by comparing the figures obtained with the bomb calorimeter. With liquid fuels the electric meter must be employed to measure the total heat production. By weighing at the beginning and at the end of a test the weight of fuel consumed is obtained.

H. F. E. H.

Apparatus for Precipitating, Filtering, and Drying in an Inert Gas. J. B. Firth and J. E. Myers.

(*Proc. Chem. Soc.*, 1911, **27**, 96.)—The apparatus designed for the above purpose is shown in the accompanying sketch.



The reacting liquids are placed in the funnels A, A, and the remainder of the apparatus is evacuated through a Büchner flask fitted to the filter-funnel H. The apparatus is then filled with the inert gas through a side-tube at K. The tap D being closed, the reacting liquids are run into the precipitating tube C, and are mixed by means of the stirrer B, which slides in a rubber stopper lubricated with rubber grease. When the reaction is complete, the tap D, which has a wide bore, is opened, and the precipitate filtered off in the funnel H. The washing and drying are carried out by drawing suitable liquids through the precipitate, and finally aspirating a stream of the inert gas to remove the liquids used for drying. There is a ground glass joint at G, by means of which the funnel is prepared for filtration and the precipitate is removed. This apparatus can be used with satisfactory results in the preparation of copper hydride.

Adaptation of the Tap-Funnel to Rapid Electro-Analysis with Stationary Electrodes. T. S. Price and A. W. T. Hyde.

(*J. Soc. Chem. Ind.*, 1911, **30**, 391-396.) — The tap-funnel may be adapted for use with stationary electrode in the deposition of copper, nickel, and zinc. The type of stationary electrode used was a cylindrical cathode of sand-blasted gauze, 45 mm. long and 32 mm. in diameter, with 4 meshes to the sq. mm., its weight being about 15 grms. The anode was a cylinder of similar gauze, 44 mm. long and 22 mm. in diameter. With this equipment a concentration of less than 1 c.c. of nitric acid per 70 c.c. of electrolyte is inadvisable, while a concentration of 2 c.c. may be used with advantage. With a sufficient amount of nitric acid previous heating of the electrolyte is unnecessary, but a final temperature of 60° to 70° C. does not prevent the deposition of the copper with 2 c.c. of nitric acid per 70 c.c. of electrolyte. Zinc may be deposited satisfactorily from the sodium sulphate, sodium acetate, acetic acid electrolyte, about 65 c.c. of glacial acetic acid per 70 c.c. of electrolyte giving the most favourable results.

After an extended experience of both stationary and rotating electrodes, the authors are still of the opinion, previously expressed by Price and Humphreys (ANALYST, 1910, **35**, 274), that rotating electrodes are to be preferred in actual practice.

G. C. J.

Sensitiveness Tests by the Impact Machine (Drop Hammer). H. Kast. (*Seventh Inter. Cong. App. Chem.*, 1909, Section III.B, **23**.) — In the author's apparatus (D.R.G.M. 281,313), made by E. A. Lentz, Berlin, the explosive is placed between the faces of two hard steel plungers of ½ sq. cm. cross-sectional area sliding in a hard steel sleeve. The arrangement is centred upright on the anvil of the impact machine, so that the weight falls truly on the upper plunger. The chief advantage of this arrangement over allowing the hammer to fall directly on the explosive is that it maintains constant the area struck. It is claimed that the apparatus meets the conditions to be fulfilled by a satisfactory impact machine, which are stated. Details of the method of using it are given. O. E. M.

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REPORTS, ETC.

Analysis of Crude Glycerol. (International Standard Methods, 1911.)—The valuation of crude glycerol has in recent years assumed greater commercial importance owing to the increased value of the commodity. The want of uniformity in the methods and processes of analyses, together with the irregularity of the results obtained, emphasised the desirability for the standardisation of crude glycerol analysis; so, with this object in view, committees were formed in America, France, Germany, and Great Britain. These committees worked, in the first instance, independently, but were ultimately brought together, and after a series of conferences the conclusions arrived at by the various committees were summarised and drawn up in the form in which they are now presented. The methods detailed in this report have the unanimous support of each of the above committees, and are strongly recommended by them as International Standards.

SAMPLING.—The most satisfactory method available for sampling crude glycerol liable to contain suspended matter, or which is liable to deposit salt on settling, is to have the glycerol sampled by a mutually approved sampler as soon as possible after it is filled into drums, but in any case before any separation of salts has taken place. In such cases he shall sample with a sectional sampler (a suitable sampling apparatus is described in an appendix to the report), then seal the drums, brand them with a number for identification, and keep a record of the brand number. The presence of any visible salt or other suspended matter is to be noted by the sampler, and a report of same made in his certificate, together with the temperature of the glycerol. Each drum must be sampled. Glycerol which has deposited salt or other matters cannot be accurately sampled from the drums, but an approximate sample can be obtained by means of the sectional sampler, which will allow a complete vertical section of the glycerol to be taken, including any deposit.

ANALYSIS.—1. *Determination of Free Caustic Alkali.*—Weigh 20 grms. of the sample into a 100 c.c. flask, dilute with approximately 50 c.c. of freshly-boiled distilled water, add an excess of neutral barium chloride solution, 1 c.c. of phenolphthaleïn solution, make up to the mark, and mix. Allow the precipitate to settle, draw off 50 c.c. of the clear liquid, and titrate with normal acid ($\frac{N}{1}$). Calculate to percentage of Na_2O existing as caustic alkali.

2.—*Determination of Ash and Total Alkalinity.*—Weigh 2 to 5 grms. of the sample in a platinum dish, burn off the glycerol over a luminous Argand burner or other source of heat giving a low flame temperature, the temperature being kept low to avoid volatilisation and the formation of sulphides. When the mass is charred to the point that water will not become coloured by soluble organic matter, lixiviate with hot distilled water, filter, wash and ignite the residue in the platinum dish. Return the filtrate and washings to the dish, evaporate, and carefully ignite without fusion. Weigh the ash.

Dissolve the ash in distilled water and titrate total alkalinity, using as indicator methyl orange cold or litmus boiling.

3. *Determination of Alkali present as Carbonate.*—Take 10 grms. of the sample, dilute with 50 c.c. distilled water, add sufficient $\frac{N}{1}$ acid to neutralise the total alkali found at (2), boil under a reflux condenser for fifteen to twenty minutes, wash down the condenser tube with distilled water, free from carbon dioxide, and titrate back with $\frac{N}{1}$ NaOH, using phenolphthaleïn as indicator. Calculate the percentage of Na_2O. Deduct the Na_2O found in (1). The difference is the percentage of Na_2O existing as carbonate.

4. *Alkali combined with Organic Acids.*—The sum of the percentages of Na_2O found at (1) and (3) deducted from the percentage found at (2) is a measure of the Na_2O or other alkali combined with organic acids.

5. *Determination of Acidity.*—Take 10 grms. of the sample, dilute with 50 c.c. of distilled water free from carbon dioxide, and titrate with $\frac{N}{1}$ NaOH and phenolphthaleïn. Express in terms of Na_2O required to neutralise 100 grms.

6. *Determination of Total Residue at* 160° *C.*—For this determination the crude glycerol should be slightly alkaline with Na_2CO_3, not exceeding the equivalent of 0·2 per cent. Na_2O, in order to prevent loss of organic acids. To avoid formation of polyglycerols, this alkalinity must not be exceeded.

Preparation of Glycerol.—Ten grms. of the sample are weighed into a 100 c.c. flask diluted with water, and the calculated quantity of $\frac{N}{1}$ HCl or Na_2CO_3 added to give the required degree of alkalinity. The flask is filled to 100 c.c., the contents mixed, and 10 c.c. measured into a weighed Petrie or similar dish 2·5 inches diameter and 0·5 inch deep, which should have a flat bottom. In the case of crude glycerols abnormally high in organic residue, a less quantity is to be evaporated, so that the weight of organic residue does not materially exceed 30 to 40 mgrms.

Evaporation of the Glycerol.—The dish is placed on a water-bath (the top of the 160° C. oven acts equally well) until most [illegible] the water has evaporated. From this point the evaporation is effected in the oven. Satisfactory results are obtained in an oven measuring 12 inches cube, having an iron plate $\frac{3}{4}$-inch thick lying on the bottom to distribute the heat. Strips of asbestos millboard are placed on a shelf halfway up the oven. On these strips the dish containing the glycerol is placed.

If the temperature of the oven has been adjusted to 160° C. with the door closed, a temperature of 130° to 140° C. can be readily maintained with the door partially open, and the glycerol, or most of it, should be evaporated off at this temperature. When only a slight vapour is seen to come off, the dish is removed and allowed to cool.

An addition of 0·5 to 1 c.c. of water is made, and by a rotary motion the residue brought wholly or nearly into solution. The dish is then allowed to remain on a water-bath or top of the oven until the excess water has evaporated and the residue is in such a condition that on returning to the oven at 160° C. it will not spit. The time taken up to this point cannot be given definitely, nor is it important. Usually two to three hours are required. From this point, however, the schedule of time must be strictly adhered to. The dish is allowed to remain in the oven, the temperature of which is carefully maintained at 160° C. for one hour, when it is removed, cooled, the residue treated with water, and the water evaporated as before. The residue is then subjected to a second baking of one hour, after which the dish is allowed to cool in a desiccator over sulphuric acid and weighed. The treatment with water, etc., is repeated until a constant loss of 1 to 1·5 mgrms. per hour is obtained.

Corrections to be Applied to the Weight of the Total Residue.—In the case of acid glycerol, a correction must be made for the alkali added. One c.c. $\frac{N}{1}$ alkali represents an addition of 0·022 grm. In the case of alkaline crudes a correction should be made for the acid added. Deduct the increase in weight due to the conversion of the $NaOH$ and Na_2CO_3 to $NaCl$. The corrected weight, multiplied by 100, gives the percentage of *total residue at* 160° *C.*

Preserve the total residue for the determination of the non-volatile acetylisable impurities.

7. *Organic Residue.*—Subtract the ash from the total residue at 160° C. Report as organic residue at 160° C. (*Note.*—It should be noted that alkaline salts of organic acids are converted to carbonates on ignition, and that the CO_3 radicle thus derived is not included in the organic residue.)

8. *Moisture.*—This test is based on the fact that glycerol can be completely freed from water by allowing it to stand *in vacuo* over sulphuric acid or phosphoric anhydride.

Two to three grms. of very pure bulky asbestos freed from acid soluble material which has been previously dried in a water-oven are placed in a small stoppered weighing-bottle of about 15 c.c. capacity. The weighing-bottle is kept in a vacuum desiccator furnished with a supply of concentrated sulphuric acid, under a pressure equivalent to 1 to 2 mm. of mercury, until constant in weight. From 1 to 1·5 grm. of the sample is then carefully dropped on the asbestos in such a way that it will be all absorbed. The weight is again taken, and the bottle replaced in the desiccator under 1 to 2 mm. pressure until constant in weight. At 15° C. the weight is constant in about forty-eight hours. At lower temperatures the test is prolonged.

The sulphuric acid in the desiccator must be frequently renewed.

Acetin Process for Glycerol Determination.—This process is the one agreed upon at a Conference of Delegates from the American, British, French, and German Committees, and has been confirmed by each of the above Committees as giving results nearer to the truth on crudes in general, and is the process to be used (if applicable) whenever only one method is employed. On pure glycerols the results are identical with those of the bichromate process. For the application of this process the crude glycerol should not contain over 50 per cent. water.

The following reagents are required:

1. *Best Acetic Anhydride.*—This should be carefully selected. A good sample must not require more than 0·1 c.c. normal NaOH for saponification of the impurities when a blank is run on 7·5 c.c. Only a slight colour should develop during digestion of the blank.

2. *Pure Fused Sodium Acetate.*—The purchased salt is again completely fused in a platinum, silica, or nickel dish, avoiding charring, powdered quickly, and kept in a stoppered bottle or in a desiccator. It is most important that the sodium acetate be anhydrous.

3. *A Solution of Sodium Hydroxide for Neutralising, of about* $\frac{N}{1}$ *Strength, Free from Carbonate.*—This can be readily made by dissolving pure sodium hydroxide in its own weight of water (preferably water free from carbon dioxide), and allowing to settle until clear, or filtering through an asbestos or paper filter. The clear solution is diluted with water free from carbon dioxide to the strength required.

4. $\frac{N}{1}$ *Sodium Hydroxide, Free from Carbonate.*—Prepared as above, and carefully standardised.

Some sodium hydroxide solutions show a marked diminution in strength after being boiled; such solutions should be rejected.

5. $\frac{N}{1}$ *Acid.*—Carefully standardised.

6. *Phenolphthaleïn Solution.*—0·5 per cent. phenolphthaleïn in alcohol and neutralise.

THE METHOD.—Into a narrow-mouthed flask (preferably round-bottomed) capacity about 120 c.c., which has been thoroughly cleaned and dried, weigh accurately and as rapidly as possible 1·25 to 1·5 grms. of the glycerol. Add first about 3 grms. of the anhydrous sodium acetate, then 7·5 c.c. of the acetic anhydride, and connect the flask with an upright Liebig condenser. For convenience the inner tube of this condenser should not be over 50 cm. long and 9 to 10 mm. inside.

The flask is connected to the condenser by either a ground glass joint (preferably) or a rubber stopper. If a rubber stopper is used, it should have had a preliminary treatment with hot acetic anhydride vapour.

Heat the contents and keep just boiling for one hour, taking precautions to prevent the salts drying on the sides of the flask.

Allow the flask to cool somewhat, and through the condenser tube add 50 c.c. of the carbon-dioxide-free distilled water, heated to about 80° C., taking care that the flask is not loosened from the condenser. The object of cooling is to avoid any sudden rush of vapours from the flask on adding the water, and to avoid breaking the flask. Time is saved by adding the water before the contents of the flask solidify, but the contents may be allowed to solidify and the test proceeded with the next day without detriment. The contents of the flask may be warmed to, but must not exceed, 80° C. until the solution is complete except a few dark flocks representing organic impurities in the crude. By giving the flask a rotatory motion, solution is more quickly effected. Cool the flask and contents without loosening from condenser. When quite cold wash down the inside of the condenser tube, detach the flask, wash off stopper or ground glass connection into the flask, and filter contents of flask through an acid-washed filter into a Jena glass flask of about 1 litre capacity. Wash thoroughly with cold distilled water free from carbon dioxide. Add 2 c.c. of phenolphthaleïn solution (F), then run in sodium hydroxide solution (C) or (D) until a faint pinkish-yellow colour appears throughout the solution. This neutralisation must be done most carefully. The alkali should be run down the sides of the flask, the contents of which are kept rapidly swirling with occasional agitation or change of motion until the solution is nearly neutralised, as indicated by the slower disappearance of the colour developed locally by the alkali running into the mixture. When this point is reached the sides of the flask are washed down with carbon-dioxide-free water and the alkali subsequently added drop by drop, mixing after each drop until the desired tint is obtained.

Now run in from a burette 50 c.c. or a calculated excess of $\frac{N}{1}$ NaOH (D), and note carefully the exact amount. Boil gently for fifteen minutes, the flask being fitted with a glass tube acting as a partial condenser; cool as quickly as possible, and titrate excess of NaOH with $\frac{N}{1}$ acid (E) until the pinkish-yellow or chosen end-point colour just remains. A further addition of the indicator at this point will cause a return of the pinkish colour; this must be neglected, and the first end-point taken.

From the $\frac{N}{1}$ NaOH consumed calculate the percentage of glycerol after making the correction for the blank test described below:

$$1 \text{ c.c. of } \tfrac{N}{1} \text{ NaOH} = 0{\cdot}03069 \text{ grm. of glycerol.}$$

The coefficient of expansion for normal solutions is approximately 0·00033 per c.c. for each degree C. A correction should be made on this account if necessary.

Blank Test.—As the acetic anhydride and sodium acetate may contain impurities which affect the result, it is necessary to make a blank test, using the same quantities of acetic anhydride and sodium acetate as in the analysis. After neutralising the acetic acid, it is not necessary to add more than 5 c.c. of the $\frac{N}{1}$ alkali (D), as that represents the excess of alkali usually left after saponification of the triacetin in the glycerol determination.

Determination of the Glycerol Value of the Acetylisable Impurities.—The total residue at 160° C. is dissolved in 1 or 2 c.c. of water, washed into a clean acetylising flask 120 c.c. capacity, and the water evaporated. Now add anhydrous sodium acetate, and proceed as in the glycerol determination before described. Calculate the result to glycerol.

Analysis of Acetic Anhydride.—Into a weighed stoppered vessel, containing 10 to 20 c.c. of water, run about 2 c.c. of the anhydride, replace stopper and weigh; allow to stand, with occasional shaking, for several hours, till all anhydride is hydrolysed; then dilute to about 200 c.c., add phenolphthaleïn, and titrate with $\frac{N}{1}$ NaOH. This gives the total acidity due to free acetic acid and acid formed from anhydride.

Into a stoppered weighing-bottle containing a known weight of recently distilled aniline (from 10 to 20 c.c.) measure about 2 c.c. of the sample, stopper, mix, allow to cool, and weigh. Wash contents into about 200 c.c. cold water, and titrate acidity as before. This yields the acidity due to the original, preformed, acetic acid plus one-half the acid due to anhydride (the other half having formed acetanilide); substract the second result from the first (both calculated for 100 grms.) and double result, obtaining c.c. $\frac{N}{1}$ NaOH per 100-grm. sample. One c.c. NaOH equals 0·0510 grm. of acetic anhydride.

Bichromate Process for Glycerol Determination. Reagents Required.—(*a*) *Pure Potassium Bichromate* powdered and dried in air free from dust or organic vapours, at 110° to 120° C. This is taken as the standard.

(*b*) *Dilute Bichromate Solution.*—7·4564 grms. of the above bichromate (*a*) are dissolved in distilled water and the solution made up to 1 litre at 15·5° C.

(*c*) *Ferrous Ammonium Sulphate.*—Dissolve 3·7282 grms. of potassium bichromate (*a*) in 50 c.c. of water. Add 50 c.c. of 50 per cent. (by volume) sulphuric acid, and to the cold undiluted solution add from a weighing bottle a moderate excess of the ferrous ammonium sulphate, and titrate back with the dilute bichromate (*b*). Calculate the value of the ferrous salt in terms of bichromate.

(*d*) *Silver Carbonate.*—This is prepared as required; for each test from 140 c.c. of 0·5 per cent. silver sulphate solution by precipitation with about 4·9 c.c. $\frac{N}{1}$ sodium carbonate solution (a little less than the calculated quantity of $\frac{N}{1}$ sodium carbonate should be used; any excess of alkali carbonate prevents rapid settling). Settle, decant, and wash once by decantation.

(*e*) *Subacetate of Lead.*—Boil a pure 10 per cent. lead acetate solution with an excess of litharge for one hour, keeping the volume constant, and filter while hot. Disregard any precipitate which subsequently forms. Preserve out of contact with carbon dioxide.

(*f*) *Potassium Ferricyanide.*—A very dilute solution containing about 0·1 per cent.

The Method.—Weigh 20 grms. of the glycerol, dilute to 250 c.c., and take 25 c.c. Add the silver carbonate, allow to stand, with occasional agitation, for about ten minutes, and add a slight excess (about 5 c.c. in most cases) of the basic lead acetate (*e*), allow to stand a few minutes, dilute with distilled water to 100 c.c., and then add 0·15 c.c. to compensate for the volume of the precipitate, mix thoroughly, filter through an air-dry filter into a suitable narrow-mouthed vessel, rejecting the first 10 c.c., and return filtrate if not clear and bright. Test a portion of the filtrate with a little basic lead acetate, which should produce no further precipitate. (In the great majority of cases 5 c.c. is ample.) Occasionally a crude glycerol will be found requiring more, and in this case another aliquot of 25 c.c. of the dilute glycerol should be taken and purified with 6 c.c. of the basic acetate. Care must be taken to avoid a marked excess of basic acetate.

Measure off 25 c.c. of the clear filtrate into a glass flask or beaker (previously cleaned with potassium bichromate and sulphuric acid). Add 12 drops of sulphuric acid (1 : 4) to precipitate the small excess of lead as sulphate. Add 3·7282 grms. of the powdered potassium bichromate (*a*). Rinse down the bichromate with 25 c.c. of water, and stand with occasional shaking until all the bichromate is dissolved (no reduction will take place).

Now add 50 c.c. of 50 per cent. sulphuric acid (by volume) and immerse the vessel in boiling water for two hours, and keep protected from dust and organic vapours, such as alcohol, until the titration is completed. Add from a weighing bottle a slight excess of the ferrous ammonium sulphate (C), making spot tests on a porcelain plate with the potassium ferricyanide (F). Titrate back with the dilute bichromate. From the amount of bichromate reduced calculate the percentage of glycerol.

1 grm. glycerol equals 7·4564 grms. bichromate.
1 ,, bichromate equals 0·13411 grm. glycerol.

Notes.—1. It is important that the concentration of acid in the oxidation mixture and the time of oxidation should be strictly adhered to.

2. Before the bichromate is added to the glycerol solution it is essential that the slight excess of lead be precipitated with sulphuric acid as stipulated in the process.

3. For "crudes" practically free from chlorides the quantity of silver carbonate may be reduced to one-fifth and the basic lead acetate to 0·5 c.c.

4. It is sometimes advisable to add a little potassium sulphate to insure a clear filtrate.

INSTRUCTIONS FOR CALCULATING ACTUAL GLYCEROL CONTENT.—1. Determine the apparent percentage of glycerol in the sample by the acetin process as described. The result will include acetylisable impurities, if any be present.

2. Determine the total residue at 160° C.

3. Determine the acetin value of the residue at (2) in terms of glycerol.

4. Deduct the result found at (3) from the percentage obtained at (1) and report this corrected figure as glycerol. If volatile acetylisable impurities are present, these are included in this figure.

NOTES AND RECOMMENDATIONS.—Experience has shown that in crude glycerol of good commercial quality the sum of water, total residue at 160° C. and corrected acetin result, comes to within 0·5 of 100. Further, in such "crudes" the bichromate result agrees with the uncorrected acetin result to within 1 per cent.

In the event of greater differences being found, impurities, such as polyglycerols or trimethylene glycol, are present. Trimethylene glycol is more volatile than glycerol; it can therefore be concentrated by fractional distillation. An approximation to the quantity can be obtained from the spread between the acetin and bichromate results of such distillates, trimethylene glycol showing by the former method 80·69 per cent., and by the latter 138·3 per cent., expressed as glycerol.

In valuing crude glycerol for certain purposes it is necessary to ascertain the approximate proportion of arsenic, sulphides, sulphites, and thiosulphates. The methods for detecting and determining these impurities have not formed the subject of this investigation.

(Signed) OTTO HEHNER, *Chairman.*
JOHN ALLAN.
JAMES B. MCARTHUR.
W. H. PHILLIPS.
P. TAINSH.
FRANK TATE.
J. L. BUCHANAN, *Hon. Secretary.*

RECOMMENDATIONS BY EXECUTIVE COMMITTEE.—If the non-volatile organic residue at 160° C. in the case of a soap lye "crude" be over 2·5 per cent.—*i.e.*, when not corrected for carbon dioxide in the ash—then the residue shall be examined by the acetin method, and any excess of glycerol found over 0·5 per cent. shall be deducted from the acetin figure.

In the case of saponification, distillation, and similar glycerol, the limit of organic residue which should be passed without further examination shall be fixed at 1 per cent. In the event of the sample containing more than 1 per cent., the organic residue must be acetylated, and any glycerol found (after making the deduction of 0·5 per cent.) shall be deducted from the percentage of glycerol found by the acetin test.

Composition of the Gases caused by Blasting in Mines. E. A. Mann. (*Report to the Government of Western Australia,* 1911.)—The operator, equipped with a Fleuss apparatus, took the samples at the face immediately after a blast. The sampling apparatus consisted of a box containing two 1,500 c.c. bottles, connected in parallel with a bellows fixed outside the box. Each bottle was fitted with two glass stop-cocks and a standard thermometer. Twelve compressions of the bellows (each 2,000 c.c.) were given. Carbon monoxide was determined by the iodine pentoxide method, carbon dioxide by the Haldane apparatus, described in his "Investigations of Mine Air," p. 100. Nitric oxide, hydrogen sulphide, and sulphur dioxide, were sometimes tested for by aspirating respectively through solutions of starch and potassium iodide, lead acetate, and bromine (weak). The sulphuric acid formed in the last case was determined as barium sulphate. The series of trials included the explosives commonly used in the district, and samples from South Africa, fired under various conditions. Carbon monoxide and nitric oxide were always found. The causes of their formation, and preventive measures, are discussed. O. E. M.

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REVIEWS.

ARSENICAL GAS-POISONING. By JOHN GLAISTER, M.D. 1908. Edinburgh: E. and S. Livingstone. 1910. Price 5s. net.

This short work by Dr. Glaister is the outcome of an investigation into the literature of poisoning by arseniuretted hydrogen consequent on two cases which came within the experience of the author. A very careful and detailed account is given of these two cases, one of which was fatal. Both of these cases resulted from the cleaning out of a retort which had been used for the manufacture of chlorine. The "manganese mud" which lay in the retort was shovelled by means of an iron shovel into a zinc galvanised iron pail, the workman having in each case to enter the retort. The arsenic present in the "manganese mud," together with the acid also present, resulted in the production of arseniuretted hydrogen by their action on the shovel and pail. The late Sir Thomas Stevenson made an investigation on behalf of he Home Office, and found that the débris from the retort, when placed in contact vith either zinc or iron, or both together, caused the evolution of arseniuretted ıydrogen. Sir Thomas Stevenson also made a toxicological analysis of the viscera ›f the fatal case, and found arsenic present in amount corresponding to 0·37 grain of ırsenious acid in the whole body.

The author gives a very interesting account of the clinical symptoms presented by the fatal case, the important ones being continued vomiting, the passage of blood in the urine, and jaundice. In spite of the most careful treatment, the symptoms continued, and hiccough, delirium, and collapse supervened before death, which occurred six days after the exposure to the poisonous atmosphere.

The case which did not prove fatal, only showed clinically nausea with weakness and general feeling of illness. There was no passage of blood in the urine, and no jaundice, both of which symptoms always make the prognosis very grave.

Dealing in detail with the possible sources of arseniuretted hydrogen and the numerous ways in which accidental poisoning may occur from inadvertent exposure to a tainted atmosphere, the author gives a full list of scientific and industrial operations in which this is especially likely to occur.

He also gives a summary of the professions and occupations in which there may be risk of exposure to arseniuretted hydrogen.

A valuable account is given of the symptoms and pathological changes occurring in arsenical gas-poisoning, together with a detailed description of the poisonous effects on animals, whilst the synoptical account of the recorded cases is of great interest and very instructive.

It is to be regretted that the author makes no mention of the part played by arseniuretted hydrogen in the gas-poisoning resulting from the storage of ferro-silicon, several fatal cases of which have occurred in recent years.

In a detailed description of the analytical methods for the detection of arsenic in toxicological investigations, rather too much importance is attached to the Gutzeit test, since there are many risks of fallacy with this test which do not obtain in some of the other more reliable methods.

At the end of the book is a bibliography of the subject.

The work is a valuable contribution to the subject of arsenical gas-poisoning, and should be carefully studied by all interested in this branch of toxicology.

W. H. Willcox.

Fibres used in Textile and Allied Industries. By C. Ainsworth Mitchell, B.A. (Oxon), F.I.C., and R. Maxwell Prideaux, F.I.C. London: Scott, Greenwood and Son. 1911. Price 7s. 6d. net.

In this work the authors have produced a laboratory manual for the examination and identification of the principal textile fibres, or, as they express it, for want of a better term, of fibres in the "isolated" condition, thus excluding those used in furriery and paper-making.

The method of treatment is concise, rather in the form of laboratory notes. Such a method necessarily tends to be empirical, and first principles are perhaps too much left to be inferred, but nothing which is noted in the way of criticism in the present review must be taken as intended to minimise the value of this work to all analysts whose practice may include the occasional investigation of fibrous materials. The microscopical, chemical, and physical characters of the individual fibres are well and adequately described, and the sixty-six illustrations, specially drawn for this book, are beyond all praise. These illustrations are all on a uniform scale to facilitate ready comparison, which, however, makes the cross-sections rather small, and the diagrammatic or exaggerated representation of characteristic features has been expressly avoided. The scope of the work comprises the animal, vegetable, and "artificial" fibres. All these are admirably described from the analyst's point of view; but the correlation between the analytical facts and their commercial and industrial significance is not always sufficiently developed. The authors quote freely from the general and periodical literature of the subject, but without criticism, and in many cases, one cannot help feeling, without personal verification. The section dealing with wool, the animal fibres, and silk, appears to have been more carefully prepared than the section dealing with the cellulose fibres. In stating (p. 29) that in chlorinised wool there is apparently no direct combination with chlorine, the authors have evidently overlooked Cross and Bevan's work on the chloro-proteins. The omission of a chapter on the adulterations of wool—*e.g.*, by mercerised jute, and, above all, the omission of a scheme for the detection and estimation of shoddy and other forms of re-manufactured wool, one of the most important and most difficult tasks of the fibre-specialist, constitutes a serious gap in this section of the book. On p. 73 the statement that artificial silk is unattacked by cuprammonium solution calls for comment. Throughout the book typographical errors are more frequent than they should be (how do the authors finally decide to spell the word "coloration"?), and on p. 96 the misplacement of the decimals is responsible for the statement that raw Egyptian cotton contains 2·50-2·56 per cent. of nitrogen. Objection may be taken to the method prescribed for the preparation of the cuprammonium reagent (p. 97), hence the not very characteristic appearance of Fig. 37. A reagent sufficiently powerful for all the purposes of the microscopist can only be obtained by the direct action of the strongest ammonia on copper turnings. For the analysis of sized cotton goods the authors describe an old method used by Thomson in 1877, but omit to say how they themselves would undertake the operation in 1911; there is no reference to the use of diastase in this connection. On p. 117 Schwalbe's toluene-distillation method is quoted for the testing of hydrated mercerised cellulose, although Schwalbe himself abandoned this method soon after its publication. The iodine colour-tests for mercerised cotton described on pp. 119-121 are of little practical value. In the chapter on artificial silks the authors have evidently had to depend too much on outside sources of information. Theoretical principles expounding the relations between the different systems adopted for the formation and twisting of the threads and the contour of the cross sections, also between the methods of dissolving the cellulose and the modified chemical properties of the products, are neglected, and the chapter becomes a compilation of empirical facts. First principles should at once have suggested to the authors that Figs. 44 and 45 have been inadvertently transposed. No reference is made to the detection and estimation of combined formaldehyde in the semi-waterproofed ("sthenosed") silks and its influence on tensile strength.

The problem of differentiating between flax and hemp might have been more clearly developed and expounded from the authors' own experience. The application of the cuprammonium reagent should have been illustrated, also the characteristic cuticular residues left after this treatment. In the succeeding chapters the following corrections should be noted: (p. 136) "cottonised flax," in the form of yarns spun like cotton from carded flax tow, is still occasionally met with in commerce; (p. 156) flax yarns, even when fully bleached, nearly always contain residues of the cortical parenchymatous tissue; (p. 163) the value of 2·0 to 2·7 mm. for the length of the ultimate fibre of Manila hemp is far too small—6 mm. would be nearer the mark. This error is quoted from Wiesner, and the authors might easily have given instead the results of their own personal measurements; a description should also have been given of the characteristic cortical tissues ("sheave") found in Manila hemp. On p. 181 African piassava is described as a single ultimate fibre, whereas a single fibro-vascular bundle is intended. Lastly, the authors might have included in the scope of their subject the "artificial horsehairs" produced by coating textile yarns with a layer of dissolved cellulose, also the textile yarns manufactured from twisted paper.

J. F. Briggs.

A Course of Practical Work in Agricultural Chemistry for Senior Students. By T. B. Wood, M.A., Drapers Professor of Agriculture in the University of Cambridge. Cambridge: At the University Press. Price 2s. 6d. net.

The contents of this pamphlet are the outcome of seventeen years' experience in teaching a class of senior students in the University of Cambridge, and the hope is expressed by the author that it may be found useful to teachers in other institutions who are attempting to give their senior students some practical acquaintance with the modern developments of agricultural chemistry. To fulfil such ambitious objects within fifty-six pages of letter-press would be a difficult task, and a perusal of the text shows it to be devoted entirely to a description of methods of analysis of soils, manures, milk, carbohydrates, proteïns, oils, food, roots, cereals, etc. Of these the methods relating to agricultural chemistry are the best, though some of them are of such a rough-and-ready character—notably those for the qualitative examination of soil—that they appeal to the untrained agriculturist rather than to the senior student. As an example of the slovenly way in which the pamphlet is written, the paragraph dealing with the estimation of nitrates in soil may be cited: "Take 1 cc. of 'soil water' in a perfectly clean test tube, add about 3 c.c. of strong H_2SO_4, shake to mix, and cool. Add about 1 c.c. of carbazol solution, shake and leave to stand five minutes. Match this colour with a solution of KNO_3 containing 10 parts N in 1,000,000, *i.e.*, 0·072 grm. KNO_3 in 1000 c.c."

Under milk analysis it is stated that there are two ways of estimating fat in milk, chemically by the Werner-Schmid method, and mechanically by the Gerber method. As a general test for the carbohydrates, the author observes that all carbohydrates, except cellulose, when boiled for some time with dilute acid, and then neutralised, give sugars which reduce Fehling's solution. He contradicts this on the next page by stating that cellulose under these circumstances is converted into glucose. The remarks on the sugars and their estimation are in many instances misleading and inaccurate.

The writer much regrets having to pass such severe strictures on a work emanating from a University which has done so much to promote agricultural science.

ARTHUR R. LING.

THE SALE OF FOOD AND DRUGS ACTS. By Sir W. J. BELL, C. F. LLOYD, and R. A. ROBINSON. Pp. xxvi and 303. London: Butterworth and Co.; Shaw and Sons. 1910. Price 8s. 6d. net.

This, the fifth, edition of the book has been enlarged so as to include the Butter and Margarine Act, 1907, and the Butter Factories Registration Order, 1907. More than fifty decisions of the different superior courts of the United Kingdom have been reported since the publication of the last edition of the book, and these, together with a number of circular letters from the various Government departments, are also included. The book contains an immense amount of information for the use of those engaged in the working of the Acts relating to the Sale of Foods, Drugs, etc., every section of the various Acts being commented upon, and the legal decisions relating to proceedings taken under the sections are recorded concisely. The information given in the volume has been brought up to date, and deals with the whole of the laws on the subject, from the Adulteration of Coffee Act, 1718, to the Fertilisers and Feeding Stuffs Regulations, 1910. An alphabetical list of cases is given and an efficient index is provided. W. P. SKERTCHLY.

MODERN INDUSTRIAL CHEMISTRY. By H. BLÜCHER. Translated by J. P. MILLINGTON, M.A., B.Sc. London: The Gresham Publishing Company. 1911. Price 30s.

In the Publishers' Note it is stated that this book is designed to cover the whole range of subjects with which the chemist and manufacturer are usually concerned, and that it is intended to occupy a position between the textbook and the lexicon. When the whole field of chemical technology is covered in something under 800 pages, it will be readily understood that the various subjects cannot have been dealt with at all fully. As a matter of fact, it is somewhat difficult to understand the precise class of person to whom this work would be likely to appeal, since the information is in the great majority of cases too incomplete to be of any value to inquirers seeking really detailed information, whilst, on the other hand, the book is not one to which the ordinary scientific reader would be likely to be attracted. On the whole, the subject-matter seems to be accurate; but some of the articles appear to have been hastily compiled, and contain information which is neither correct nor up to date, whilst in other cases the processes (*e.g.*, brewing) dealt with have special reference to German procedure, and are, therefore, of less interest to the English reader. The list of firms given in connection with the various chemical products dealt with may sometimes be useful, but they have rather the appearance of advertisements, and in certain cases the firms mentioned are scarcely representative ones. The extent to which condensation has been carried out may be gathered from the fact that the technology of sodium and its various compounds are dealt with in some seventeen pages. The book is illustrated, and some of the illustrations are clear and well chosen, and the printing and general style of the book are good. A. CHASTON CHAPMAN.

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NEW BOOKS.

ORGANIC CHEMISTRY FOR THE LABORATORY. By W. A. NOYES, Ph.D. 8vo. volume. The Chemical Publishing Company: Easton, Pa., United States of America. 1911. Price $2.

A second edition, revised and enlarged, contains 273 pages of subject-matter, with 41 illustrations, and an alphabetical index of subjects. The subject-matter is classified under the following heads: I. Analysis of Compounds of Carbon. II. General Operations. III. Hydrocarbons. IV. Alcohols and Phenols. V. Ethers. VI. Aldehydes, Ketones, and their Derivatives. VII. Acids. VIII. Derivatives of Acids. IX. Hydroxy- and Ketonic Acids. X. Carbohydrates. XI. Halogen Compounds. XII. Nitro Compounds. XIII. Amines. XIV. Diazo-, Hydrazo-, Nitroso-, and other Nitrogen Compounds. XV. Sulphur Compounds. XVI. Qualitative Examination of Carbon Compounds. Reagents.

YEAR-BOOK OF PHARMACY AND TRANSACTIONS OF THE BRITISH PHARMACEUTICAL CONFERENCE. Edited by J. O. BRAITHWAITE. 8vo. volume. London: J. and A. Churchill. 1910. Price 10s. net.

This year-book comprises abstracts of papers relating to Pharmacy, Materia Medica, and Chemistry, contributed to British and foreign journals from July, 1909, to June, 1910; and the Transactions of the British Pharmaceutical Conference, which was held at Cambridge in July, 1910.

JULY, 1911. Vol. XXXVI., No. 424.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The ordinary meeting of the Society was held on Wednesday evening, May 31, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. W. Voelcker, A.R.S.M., F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. H. C. Huish was read for the second time; and certificates in favour of Messrs. Ronald Devereux Carty, A.R.C.Sc.I., A.I.C., The Laboratories, Survey Department, Public Works Ministry Gardens, Cairo, assistant in the Survey Department's Laboratory, Cairo; William Keighley Walton, "Beechwood," Upper Rushton Road, Thornbury, Bradford, assistant to Messrs. Richardson and Jaffé; and G. F. Wesley-Martin, Castle Park, Lancaster, chemist to Messrs. Maudsley and Son, were read for the first time.

Mr. A. D. Heywood, A.I.C., was elected a member of the Society.

The following papers were read: "Notes on the Analysis of Margarine," by Cecil H. Cribb, B.Sc., F.I.C., and P. A. Ellis Richards, F.I.C.; "Observations on Some Methods of estimating Cocoanut Oil and Butter in Butter and Margarine," by Cecil Revis and E. Richards Bolton; "The Composition of Milk," by H. Droop Richmond, F.I.C.; "The Estimation of Quinine as the Acid Citrate in Certain Organic Liquids," by T. Cockburn, F.I.C., and J. W. Black; "The Determination of the Amount of Dissolved Oxygen absorbed by Sewage Effluents containing Nitrite, and of the Amount of Nitrite in Sewage Effluents or Water," by R. W. Clarke, F.I.C.; and "Further Analyses of Ghee," by Cecil Revis and E. Richards Bolton.

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NOTES ON THE ANALYSIS OF MARGARINE.

By CECIL H. CRIBB, B.Sc., F.I.C., and P. A. ELLIS RICHARDS, F.I.C.

(*Read at the Meeting, May* 31, 1911.)

The frequent occurrence of cocoanut oil in margarine mixtures (butter substitutes) at the present time has introduced a new factor into butter analysis. It is fully recognised that the simple method of examination by the official Reichert-Wollny process is no longer effective for determining whether margarine comes within the legal definition or not. Still less is it possible in the presence of cocoanut oil to arrive at the proportions in a mixture by this method.

Polenske's process and its modifications afford valuable assistance in this direction, more especially where only butter and cocoanut oil are present. Such mixtures, however, are the exception rather than the rule. Usually when margarine contains cocoanut oil it also contains animal fats other than butter, and it is precisely in cases such as these that difficulties arise.

Up to the present papers on the Polenske process and its modifications have largely dealt with the analysis of mixtures of two constituents, of which cocoanut oil is one. A number of processes, mostly modifications of the original Polenske, have been brought forward, but few, if any, of them offer any real advantage over the original method, and some at least greatly increase the time and trouble involved without materially improving the results. Ample illustration of this is afforded by Shrewsbury and Knapp (ANALYST, 1910, **35**, 385), and other authors, who show that the differences between the figures for cocoanut oil and butter-fat, upon which calculations are based, are not so great as in the Polenske process. The Polenske figure for cocoanut oil (17·5) is between five and six times that of butter (2 to 3·5), and, as far as we are aware, none of these other processes will improve on this ratio. It is obvious that the Wollny and Polenske processes together enable us to obtain figures from which it should be possible to calculate the percentages of butter and cocoanut oil in a mixture, leaving the third constituent, if present, to be calculated by difference, but no one has hitherto pointed out how this is to be done.

As the Wollny process is already in universal use, it would be very advantageous if a combination of the two were adopted as the basis of any new official method to be recommended by the Butter Committee. The initial procedure in both cases is practically identical, the Polenske being really an extension of the original Wollny method. To render the combined processes effective for the purposes we suggest, it is necessary to ascertain how far, if at all, the presence of one fat in a mixture affects the Wollny-Polenske figure of another.

It has been already shown by various workers that in mixtures consisting of *cocoanut oil and butter* ONLY, the proportion of the constituents can be determined with approximate accuracy by the simple Polenske process, and our experience confirms this; but the results obviously leave room for considerable improvement (see Table I., p. 331).[*]

The method of calculating the composition of the mixtures is as follows:

From the volatile insoluble acids we deduct one-tenth of the volatile soluble acids, assuming that this amount is derived from the butter present. This is not strictly correct, as a proportion of the soluble volatile acids is really due to the cocoanut oil. The residual volatile insoluble acids are calculated into terms of cocoanut oil on the assumption that genuine cocoanut oil yields 17·5 c.c. volatile insoluble acids. It is obvious from the table that the percentages of cocoanut oil obtained are low to the extent of from 2·5 to 9 per cent., the butter, if calculated by difference, being in excess to a corresponding extent. If any attempt be made to calculate the butter from the soluble volatile acids, the possible error is much greater owing to the wide variations in the soluble volatile acids of this fat. Even with cocoanut oil some disturbing influences are evidently at work, and it is to a consideration of these that our attention has been mainly directed.

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[*] The figures in the table were obtained by saponifying 5 grms. of the fat with 20 c.c. of glycerol soda, employing the Wollny-Reichert process and apparatus throughout, but with powdered pumice (90 mesh). The distillate was cooled to 15 C. instead of 12 C., and the insoluble volatile acids collected as in the Polenske process.

In the first place the volatile soluble acids have to be corrected (*a*) for the proportion of soluble volatile acids due to the cocoanut oil, and (*b*) for the proportion of the insoluble volatile acids derived from the cocoanut oil becoming dissolved owing to the fact that while the volume of the distillate remains constant the volatile "insoluble" acids diminish directly with the cocoanut oil. In the second place the insoluble volatile acids have (*c*) to be increased by the amount that has been deducted from the soluble volatile acids, and (*d*) to be diminished by the amount of insoluble volatile acids due to the butter present.

In addition to the above, some experiments by Tatlock and Thomson (*J. Soc. Chem. Ind.*, 1909, **28**, 69-72; ANALYST, 1909, **34**, 223) suggest the necessity of a further correction—namely, for the retention of both soluble and insoluble volatile acids by the non-volatile fatty acids of butter, so that a portion of the volatile acids fails to reach the distillate.

We now propose to consider these various corrections in detail.

1. (*a*) *Correction of Volatile Soluble Acids for those due to Cocoanut Oil.*—The volatile soluble acid figure is derived both from the butter and the cocoanut oil. To ascertain the amount due to butter alone the number of c.c. due to cocoanut oil must, of course, be deducted. As cocoanut oil gives a fairly constant and small figure of 6·5 (for 5 grms. of fat) it is only necessary to know approximately the proportion of this fat in order to make the correction. For this purpose it is sufficient in the first instance to deduct from the volatile insoluble acids a number of c.c. equivalent to one-tenth of the volatile soluble figure, and to calculate the rest into cocoanut oil on the assumption that 17·5 c.c. corresponds to 100 per cent. of cocoanut oil. From the percentage thus obtained the soluble volatile acid figure for the cocoanut oil thus found is calculated and deducted from the total volatile soluble figure. The remainder, subject possibly to further correction, represents in c.c. the volatile soluble acids due to the butter present. As the matter is somewhat involved, a concrete example may be taken: 5 grms. of a mixture of butter-fat and cocoanut oil are found to give—

Soluble volatile acids, 25·2 c.c. Polenske figure, 5·4 c.c.

From the Polenske figure one-tenth of 25·2, *i.e.*, 2.5, is deducted. The remainder, 2·9, is equivalent to 16·5 per cent. of cocoanut oil, which should yield 1·07 c.c. of volatile soluble acids. This is then subtracted from 25·2, the result being 24·13 c.c. The volatile soluble figure for the butter employed in this mixture was 29·6, and calculating from this, the butter would be estimated at 81 per cent. The actual proportions taken were: Cocoanut oil, 20 parts; Butter-fat, 80 parts. Of course with a butter deviating more widely from the average, the figure for the butter would not be so good.

(*b*) *Correction for Solubility of So-called Insoluble Volatile Acids.*—Further corrections, however, as we have already suggested, must be made—namely, for the solubility in water of the so-called insoluble acids derived from the cocoanut oil.

From various experiments we have been led to adopt 0·8 c.c. as the figure to be deducted from the soluble acids and added to the insoluble acids. It must be borne in mind, however, that this figure is largely dependent on the temperature at which the distillate is filtered. In view of the great inconvenience of having to cool the latter to as low a temperature as 12° C., we have adopted 15° C., as it is only in the height of summer that the water-supply is likely to be warmer than this. If the correction be made, it becomes necessary to alter the figure for the cocoanut oil insoluble acids, on which the calculation of the percentage of the cocoanut oil is ultimately based, from 17·5 to 18·3 c.c. If this be done, the percentage of cocoanut oil becomes 20·2, as compared with 20 actually present. This correction also affects the percentage of butter, inasmuch as the soluble volatile acids from which this is calculated are further reduced. After the necessary allowance has been made, the proportion of butter becomes 79·9 instead of 80.

Table I. (p. 331) gives the results of the analyses of several mixtures of known composition, and shows also the effect of the various corrections. In view of the fact of the wide variations in the volatile soluble acids of different butters, it would be more satisfactory to calculate the butter by difference.

The presence of a third constituent seriously affects the complexity of the problem. It is hardly necessary to point out that the third fat will also contribute its quota of soluble and insoluble volatile fatty acids, and, for this, allowance must be made. In addition, other and less obvious effects are produced by the presence of the third constituent. Both butter-fat and margarine-fat exercise a retentive influence on the distillation of the soluble and insoluble volatile acids.

Of much more importance, however, is the fact that the whole process is an empirical one, and assumes a more or less definite ratio between the quantity of the volatile fatty acids and the volume of water distilled, and any departure from this alters the composition of the distillate. This has already been pointed out by Tatlock and Thomson (*loc. cit.*), but hitherto, so far as we are aware, the fact has not received any special attention.

Taking respectively 5 grms. and 1 grm. of cocoanut oil and distilling in each case in the usual way, there is a wide disproportion between the volume of the distillate and the weight of fatty acids distilled.

A series of experiments of our own illustrate this fact, and we found, as has been already demonstrated by Tatlock and Thomson, that in the case of both butter-fat and cocoanut oil, the soluble and insoluble volatile acids are quite out of proportion to the weights of the respective fat taken, increasing relatively as the quantity of the latter diminishes.

The retentive influence of the non-volatile fatty acids of margarine and butter upon the volatile acids of cocoanut oil and butter is shown by the results of some experiments given in Table II. (p. 332), in which the washed insoluble non-volatile acids from 5 grms. of certain fats were added to the liberated fatty acids of the same or other fats and distilled in the usual way.

In interpreting the figures, it should be borne in mind that the extra portion of insoluble acids added to the contents of the flask gives both soluble and insoluble acids as shown in Table III. (p. 332).

If allowance be made for this, the fact is obvious that the volatile acids are held back under these conditions. Thus, taking the Experiment 2, Table II., the figure for the volatile soluble acids is 28·75 for the butter-fat, and 0·2 for the margarine fatty acids added. The figure obtained was only 27·1—a loss of 1·85 c.c. In the case of the insoluble acids the calculated figure was 3·1, as compared with 2·0 actually found. To get the best results from the process in the case of a mixture of more than two fats, some correction must be made for these phenomena.

In Table IV. (p. 333) we have given the analytical results obtained by the examination of a series of known mixtures, the butter-fat content varying between 5 and 15 per cent.

We have not yet succeeded in working out a satisfactory method for correcting the figures obtained for the combined effect of the retention of volatile acid and the variation in the ratio between the water and the quantity of acids distilled, but a consideration of the figures in the table shows that an addition of 1·9 c.c. to the insoluble volatile acids, and a corresponding reduction of the same amount from the soluble volatile acids gives very fair results.

It will be observed that we have applied a different correction in the case of the three constituent mixtures from that used where butter and cocoanut oil only are present, and some justification for doing so is to be found in the fact that margarine fat appears to exert a much greater retentive influence on the volatile acids than does butter-fat (Table V., p. 333).

Although the suggestions put forward are only tentative ones, we trust that it may be of some assistance to the Butter Committee in the investigation upon which they are now engaged.

TABLE I.

	I.	II.	III.	IV.
Soluble volatile acids found	27·3	25·2	20·7	16·1
Insoluble " "	4·0	5·4	8·3	10·2
Cocoanut oil present	10	20	40	50
(*a*) Deducting one-tenth soluble volatile figure from the insoluble acids ...	7·5	16·5	35·5	49·1
(*b*) Ditto + correction for solubility of insoluble acids of cocoanut oil (0·8 and 18·3)	11·4	20·2	38·2	51·3
Butter-fat present	90	80	60	50
(*c*) From soluble volatile acids—ditto for cocoanut oil found in (*b*) (29 figure)	91·7	82·4	62·4	44·8
(*d*) From ditto, less also 0·8 c.c. for solubility of so-called "insoluble" acids...	89·0	80·3	60·0	48·6
(*e*) By difference from cocoanut oil found—				
difference from (*a*)	92·5	83·5	64·5	50·9
difference from (*b*)	or 88·6	or 79·8	or 61·8	or 48·7

Table II.

	Volatile Soluble Acids.		Insoluble Acids.		Loss of Acids.	
	Found.	Calculated.	Found.	Calculated.	Soluble.	Insoluble.
(1) 5 grms. butter-fat + insoluble acids from another 5 grms.	27·5	30·25	2·9	4·3	2·70	1·4
(2) 5 grms. butter-fat + insoluble acids from 5 grms. margarine (free from cocoanut oil)...	27·1	28·95	2·0	3·1	1·85	1·1
(3) 5 grms. cocoanut oil + insoluble acids of 5 grms. cottonseed oil	5·6	6·40	14·7	17·5	0·80	2·8
(4) 5 grms. cocoanut oil + insoluble acids of 5 grms. butter-fat	6·5	9·20	11·0	17·7	2·70	6·7

Table III.

Fat taken.	Volatile Soluble Acids.	Volatile Insoluble Acids.	Observations.
5 grms. cocoanut oil:			In each case the residual fatty acids left in the flask were well washed with water; and then redistilled with the usual volume of acidulated water.
1st distillation ...	6·40	17·40	
2nd ,, ...	3·20	9·60	
3rd ,, ...	2·10	7·60	
4th ,, ...	1·40	6·20	
5th ,, ...	0·80	5·50	
5 grms. butter-fat:			The volatile insoluble acids of cocoanut oil were liquid in the first distillate, semi-solid in the second, and quite solid in the succeeding distillates.
1st distillation ...	28·50	2·70	
2nd ,, ...	1·50	1·70	
3rd ,, ...	0·65	1·35	
4th ,, ...	0·65	1·15	
5 grms. margarine:			
1st distillation ...	0·50	0·50	
2nd ,, ...	0·20	0·30	

TABLE IV.

(Calculations based on corrected figures—viz., 1·9 minus $\frac{1}{10}$ volatile soluble figure added to insoluble acids, and 1·9 + cocoanut oil soluble volatile acids deducted from soluble volatile figure.)

Volatile Soluble Acids.		Volatile Insoluble Acids.		Cocoanut Oil.		Butter-Fat.		Margarine-Fat.	
Found.	Corrected.	Found.	Corrected.	Taken.	Found.	Taken.	Found.	Taken.	Found.
5·8	1·70	5·0	6·3	35	34·5	5	5·8	60	59·7
6·2	2·70	3·3	4·5	25	24·5	10	9·3	65	66·2
7·0	3·30	4·3	5·5	30	30·0	10	11·4	60	58·6
7·8	3·30	6·0	7·2	40	39·4	10	11·4	50	49·2
8·0	4·15	4·4	5·5	30	30·0	15	14·3	55	55·7

TABLE V.

Cocoanut Oil.	Butter-Fat.	Margarine-Fat.	Volatile Soluble Acids.	Volatile Insoluble Acids.	Calculated Figures.	
					Volatile Soluble Acids.	Volatile Insoluble Acids.
1	4	—	25·2	5·4	22·9	5·5
1	—	4	3·6	2·8	1·7	3·9
1.5	—	3·5	4·2	4·0	2·3	5·5
1·5	3·5	—	21·4	7·0	20·8	7·1
2	3	—	20·7	8·3	18·8	8·5
2	—	3	4·9	6·7	4·2	7·6

* * * * *

OBSERVATIONS ON SOME METHODS OF ESTIMATING COCOANUT OIL AND BUTTER IN BUTTER AND MARGARINE.

By CECIL REVIS AND E. RICHARDS BOLTON.

(Read at the Meeting, May 31, 1911.)

SINCE the day of Polenske's classical work on the estimation of cocoanut oil in butter, innumerable methods have been put forward for a similar purpose; it is, however, unfortunate that nearly all the later experimenters entirely fail to realise the fact which Polenske emphasised—namely, that there is no such thing as "average butter." The failure to recognise this practically renders all these later methods of no real value. The apparent reason for this diligent search for improvement can only be ascribed to a failure to realise the scrupulous attention to experimental details which the Polenske method necessitates, and at the outset we would strongly reiterate our conviction, which will be supported by all those who observe the necessary attention to detail, that the method fully justifies its claim, and is the only chemical method by which small quantities of cocoanut oil can be detected and approximately estimated in butter. Since the method was published we have applied it to a very large number of butters of known origin, and also to admixtures of these butters with known quantities of cocoanut oil, and have only once found cocoanut oil indicated when not present, and only once failed to find it when the percentage did not fall below five.

In the case of margarine, the increasing and almost universal use of cocoanut and palm kernel oil must be our sole apology for drawing attention again to the Polenske process, and the necessity for standard conditions.

These conditions as originally laid down by Polenske are complete except in one particular (to which Mr. Richmond drew our attention, and to whom our thanks are due for the suggestion). This particular is the temperature of the still-head during distillation, as undoubtedly the composition of the distillate varies with this temperature; we have therefore carefully investigated the effect of maintaining a constant temperature in the still-head, and compared it with our ordinary conditions of work.

For this purpose we employed an apparatus similar to that used by Richmond in his studies on Steam Distillation (ANALYST, 1908, **33**, 305), and as a result (see Table IV., p. 340) we find that, so long as reasonable care is taken to protect the still-head from draught, no error arises from this source ; and as the maintenance of a high temperature depresses both the Reichert-Meissl and Polenske values, there is no advantage in employing a specially protected high-temperature still-head, beyond the fact that time, and to a certain extent the type of distillation, become of less importance.

Besides the Polenske process, we have made use of the recently published method of Shrewsbury and Knapp (ANALYST, 1910, **35**, 385), and to a limited extent of the cadmium method of Paul and Amberger (*Zeitsch. Untersuch. Nahr. Genussm.*, 1909, **17**, 1), and also the method of Kirschner (*ibid.*, 1905, **9**, 65), as being representative of the different types of methods which have been put forward. The cadmium method, on account of its extreme laboriousness and questionable value (in our hands), was soon abandoned.

The method described by Shrewsbury and Knapp, is obviously open to several sources of error; and, in spite of the favourable results obtained by others, we found ourselves quite unable to obtain concordant results with the same samples: we therefore slightly modified the method, in order to avoid as far as possible these sources of error, though at the same time the time of operation was necessarily increased.

The method as modified by us is as follows: Five grms. of the fat are saponified in the usual manner, with glycerol soda. To the soap are added 150 c.c. of boiling water, and then 5 c.c. of sulphuric acid (1 in 5). The flask is closed with an india-rubber stopper, and the whole vigorously shaken for about half a minute, cooled by standing in a stream of running water, and the liquid poured off from the cake of fatty acids through a filter as completely as possible. To the fatty acids in the flask 20 c.c. of boiling water are added, and the whole well shaken. The flask is cooled, and the water poured off through the filter as before. The solid cake is then washed with 20 c.c. of cold water, which is poured on the filter and then with a final 10 c.c. The flask is inverted over the filter till the cake is thoroughly drained. The filter-paper is then dropped into the flask containing the cake of fatty acids, 100 c.c. of 60 per cent. alcohol (by volume) added, and the whole allowed to boil under a reflux condenser with occasional shaking for half an hour. The flask is removed, cooled rapidly by shaking under a water-tap, and finally allowed to stand in water at 60° F. for half an hour. The liquid is filtered off, and 50 c.c. titrated hot with $\frac{N}{10}$ soda. The number of c.c. so obtained constitutes the modified Shrewsbury and Knapp value.

The figures obtained by us for butters with this modified method show that the information given is not so reliable as with the Polenske process (see Table I., p. 338). In the case of margarines containing cocoanut oil, the values, generally speaking, are independent of the presence of butter-fat, but the results are of less value than those obtained by the Kirschner process.

It was soon apparent to us that, while Kirschner's extension of the Reichert-Meissl process offers little value over the Polenske method for cocoanut oil in butter, it is of decided value in the case of addition to butter of margarine mixtures containing cocoanut oil, and of unique value for the examination of margarines containing cocoanut oil with or without admixture with butter; and it is our conviction that it is the only published method at present available which will decide whether butter-fat is present in a margarine containing cocoanut oil, even if the amount be only 2 per cent. in conjunction with 75 per cent. or more of cocoanut oil.

We have examined by this process is a comprehensive series of margarines containing percentages of cocoanut oil from 5 to 75 per cent., with and without the addition of from 2 to 10 per cent. of butter-fat.

As, however, these values only apply when the Reichert-Meissl-Polenske-Kirschner process is carried out under the conditions which we employed, we append for convenience *exact* details of the methods: Five grms. of the fat and 20 grms. of glycerol are weighed with a 300 c.c. flask, and 2 c.c. of 50 per cent. sodium hydroxide solution added. The flask is heated over a flame with constant shaking till it clears suddenly, the soap is then cooled and 100 c.c. of recently well boiled distilled water is added, until solution of the soap is effected. 0·1 grm. of powdered pumice sieved through butter-muslin is added, and then 40 c.c. of sulphuric acid solution (20 to 25 c.c. of strong sulphuric acid diluted to 1,000 c.c., and the solution adjusted so that 35 c.c. neutralise 2 c.c. of the sodium hydroxide solution). The flask is at once connected with the condenser, and heated with a small flame till the insoluble acids are completely melted; the flame is then increased, and 110 c.c. distilled in nineteen to twenty-one minutes. The temperature of the condenser water should be from 18° to 20° C., and the dimensions of the apparatus are to be the same as given by Polenske. When 110 c.c. have distilled, the flame is removed, and a

25 c.c. cylinder placed under the condenser to catch any drops. The 110 c.c. flask and contents are stood in water at 10° to 15° C. for fifteen minutes. After mixing the contents of the 110 c.c. flask, they are filtered and 100 c.c. titrated with $\frac{N}{10}$ baryta, using 0·5 c.c. of a 1 per cent. solution of phenolphthaleïn as indicator. This number of c.c. increased by one-tenth, after subtraction of the blank (which must be determined in an exactly similar way, by using all the reagents except the fat), is the Reichert-Meissl value. The condenser, cylinder, and 110 c.c. receiver, are washed with 18 c.c. of cold water, which are then poured over the filter used to filter the distillate, and rejected. The condenser is washed out with four successive portions of 10 c.c. of neutral alcohol, which are received in the cylinder and poured over the filter into the 110 c.c. flask, the mixed alcohol solutions being then titrated with $\frac{N}{10}$ baryta, using phenolphthaleïn as an indicator. A blank value is obtained in a similar way. The number of c.c. of $\frac{N}{10}$ baryta used, less the number used for the blank, is the Polenske figure. Rather varying results have been obtained by various observers, but the following table will be found a very fair guide:

Reichert-Meissl Values.	New Butter Values (Polenske).
32	3·5
31	3·2
30	3·0
29	2·9
28	2·7
27	2·4
26	2·0
25	1·8
24	1·7
23	1·6

A "new butter value" (Polenske) exceeding by 0·5 c.c. the figure corresponding with the Reichert-Meissl value found indicates the presence of cocoanut or palm kernel oil or their products.

Kirschner's Extension.—To the 100 c.c. of the 110 c.c. distilled and titrated with baryta (care having been taken not to exceed the neutral point) is added 0·5 grm. of finely powdered silver sulphate, and the whole allowed to stand for an hour, with occasional shaking. The liquid is then filtered, 100 c.c. measured off, 35 c.c. of water and 10 c.c. of sulphuric acid (as previously employed) added, together with a long piece of aluminium wire, and 110 c.c. again distilled off in the standard Reichert-Polenske apparatus in twenty minutes; 100 c.c. are titrated, and the number of c.c. so obtained corrected for the blank, is calculated to the Kirschner value by the following formula:

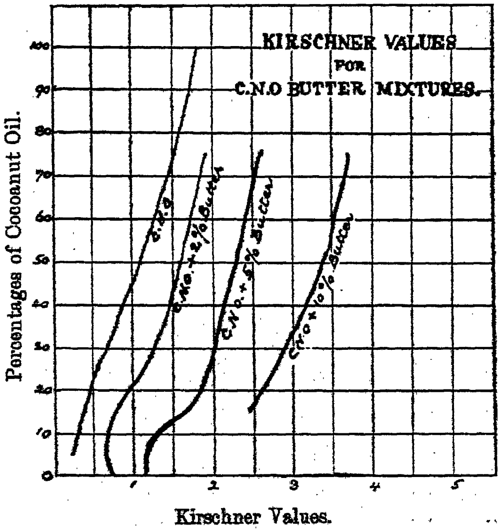
$$K = x \times \frac{121 \times (100 + y)}{10{,}000};$$

where x = the corrected Kirschner titration;
y = the number of c.c. of baryta used to neutralise 100 c.c. of the Reichert-Meissl distillate.

It will be seen on examination of Table I. (p. 338) that the variation between the Kirschner and Polenske values is greater than the variation between the

Polenske and the Reichert-Meissl values. The Kirschner value therefore assumes a certain amount of importance in the cocoanut oil butter mixtures; as will be seen, this importance is still further enhanced when the cocoanut oil is introduced into the butter as the constituent of a margarine.

This is due to the fact that the Kirschner value is practically a measure of the butyric acid content of the mixture, and is free from the difficulty which attaches to the value obtained by some of the other silver methods, in which the effect on the solubility of the silver salts of a common ion does not always seem to have been fully recognised. If the values given in Table II. are plotted, using percentages of cocoanut oil as ordinates and corresponding Kirschner values as abscissæ, it will be found that in the case of butter-free margarines containing cocoanut oil, practically a straight-line curve results, and that this curve lies wholly within that connecting the similar values obtained when 2 per cent. of butter-fat is present.



Objection may be made that constant values have been assumed for cocoanut oil, but in our experience the assumption is sufficient for the purpose. The variations of pure cocoanut oil and of the substances included under that designation are not great enough to invalidate the statements we have made. The curves for larger amounts of butter-fat lie still farther away. By means of these curves it can be at once determined from any Kirschner value, whether butter-fat is, or is not present, taking into account the corresponding Reichert-Meissl and Polenske figures. Further, for butter-free cocoanut oil margarines the Kirschner value is in complete agreement with the saponification and other values, usually concurrently determined, and so proving a most useful additional check.

Table III. (p. 340) gives the Shrewsbury and Knapp values for the same mixtures.

TABLE I.

Sample.	Reichert-Meissl.	Polenske.	Polenske Limit for Butter. +0·5 c.c.	Kirschner.	Modified Shrewsbury.
Butter:					
Irish	26·8	2·1	2·8	—	—
Danish	30·7	2·3	3·7	—	—
Irish	24·6	1·7	2·3	—	—
Irish + 5 per cent. cocoanut oil	24·1	2·3	2·2	—	—
Danish	31·9	3·4	4·0	—	—
Danish + 5 per cent. cocoanut oil	31·1	3·9	3·7	—	—
Danish	29·1	2·6	3·5	23·7	50·1
Danish + 10 per cent. cocoanut oil	27·3	4·1	3·0	21·3	70·7
French	28·3	2·8	3·3	20·1	30·1
French + 5 per cent. cocoanut oil	27·6	3·1	3·0	18·8	34·6
Irish	22·9	1·9	2·1	17·7	25·3
Irish + 10 per cent. cocoanut oil	21·7	3·3	2·0	16·5	33·9
Pure butter (source unknown):					
No. 1 butter + 5 per cent. cocoanut oil	23·5	2·5	2·1	16·6	26·6
No. 2 butter + 10 per cent. cocoanut oil ...	26·4	3·5	2·7	18·1	38·1
French	30·4	2·4	3·6	21·2	—
„	30·9	2·5	3·7	21·9	—
English	28·7	2·2	3·3	20·2	—
„	30·7	2·7	3·6	21·3	—
New Zealand	32·4	2·5	4·0	22·7	—
„	30·7	2·4	3·6	21·9	—
Danish	31·3	2·9	3·8	21·4	—
Pure Blends:					
No. 1	29·0	2·3	3·4	20·6	—
No. 2	28·6	2·2	3·3	20·4	—
No. 3	30·3	2·4	3·5	21·5	—
No. 4	29·3	2·4	3·4	21·0	—
Mixtures:					
2 per cent. cocoanut oil 6 „ oleo ... 92 „ butter ...	27·6	2·4	3·0	19·5	—
5 „ cocoanut oil 15 „ oleo ... 80 „ butter ...	24·7	2·4	2·2	17·2	—
10 „ cocoanut oil 30 „ oleo ... 60 „ butter ...	19·6	2·5	—	13·7	—

TABLE II.

Cocoanut Oil.		Butter-Fat.			
		0·0 per Cent.	2·0 per Cent.	5·0 per Cent.	10 per Cent.
0 per cent.	Reichert-Meissl ...	0·19	0·72	1·25	2·25
	Polenske	0·31	0 46	0·48	0·54
	Kirschner	0·10	0·75	1·13	1·46
5 per cent.	Reichert-Meissl ...	0·87	2·33	—	—
	Polenske	0·71	0·71	—	—
	Kirschner	0·22	0·63	—	—
10 per cent.	Reichert-Meissl ...	1·64	—	2·70	—
	Polenske	1·17	—	1·27	—
	Kirschner	0·32	—	1·27	—
15 per cent.	Reichert-Meissl ...	2·67	3·29	4·27	5·08
	Polenske	1·73	1·74	1·69	1·84
	Kirschner	0·38	0·76	1·69	2·45
25 per cent.	Reichert-Meissl ...	3·91	4·85	5·53	6·81
	Polenske	2·91	2·61	2·96	3·16
	Kirschner	0·54	1·15	1·94	2·77
50 per cent.	Reichert-Meissl ...	6·05	6·60	7·70	9·10
	Polenske	7·06	7·16	6·90	7·30
	Kirschner	1·10	1·56	2·27	3·37
75 per cent.	Reichert-Meissl ...	6·97	8·34	9·20	10·50
	Polenske	12·36	11·91	12·20	12·30
	Kirschner	1·50	1·91	2·57	3·68
100 per cent.	Reichert-Meissl ...	7·48	—	—	—
	Polenske	16·45	—	—	—
	Kirschner	1·80	—	—	—

Two different margarine bases were used, made from known pure materials:

	I. Per Cent.	II. Per Cent.
"Oleo Oil"	45	—
Lard	30	—
"Oleo Stearine"	10	25
Cottonseed oil	15	35
Soya oil	—	40

Both these margarine bases had identical Reichert-Meissl and Polenske values. It is noteworthy, however, that the modified Shrewsbury and Knapp value for I. was 13·4, and II. 22·7.

TABLE III.—MODIFIED SHREWSBURY AND KNAPP VALUES.

Cocoanut Oil.	Butter-Fat.			
	0·0 per Cent.	2·0 per Cent.	5 per Cent.	10 per Cent.
0 per cent. ...	13·4	13·8	13·4	15·1
5 ,, ...	18·1	15·9	—	—
10 ,, ...	20·1	—	21·6	—
15 ,, ...	23·3	26·4	26·6	24·7
25 ,, ...	39·6	64·0	44·6	60·8
50 ,, ...	98·1	136·7	112·4	139·1
75 ,, ...	190·6	198·1	192·6	196·8

TABLE IV.—COMPARISON OF STANDARD AND RICHMOND'S POLENSKE APPARATUS.

Sample.	Standard.		Richmond.	
	Reichert-Meissl.	Polenske.	Reichert-Meissl.	Polenske.
Butter:				
Irish	26·8	2·1	26·8	2·0
Danish	30·7	2·3	30·3	2·1
Irish	24·6	1·7	24·2	1·6
Irish + 5 per cent. cocoanut oil ...	24·1	2·3	23·7	2·2
Danish	29·1	2·6	28·7	2·3
Danish + 10 per cent. cocoanut oil	27·3	4·1	27·0	3·8
French	28·3	2·8	27·8	2·7
Irish + 10 per cent. cocoanut oil	21·7	3·3	21·4	3·2
Margarine + 5 per cent. butter (Irish)	1·25	0·48	1·16	0·36
Margarine + 10 per cent. butter ...	2·25	0·54	2·22	0·46
Margarine + 25 per cent. cocoanut oil	3·91	2·91	3·73	2·71
Margarine + 25 per cent. cocoanut oil + 10 per cent. butter	6·81	3·16	6·76	3·06

DISCUSSION.

The PRESIDENT, in inviting discussion, remarked that in these empirical methods it was important that the prescribed conditions of working should always be faithfully adhered to. Even the use of an upright instead of an inclined condenser, or of pumice of varying degrees of fineness, would cause the results to vary considerably.

Mr. RICHMOND said that, in attempting to ascertain the effect of different constituents on the proportion of soluble and insoluble volatile acids obtained on distillation, Messrs. Cribb and Richards had set themselves a very large problem, the distillation of butter-fat being one of the most complicated cases of steam distillation, since the water-soluble acids (which included both the soluble and insoluble volatile acids) were being distilled from a continually diminishing quantity of water, and were also soluble in a different ratio in the fatty acids, which latter were also slowly diminishing in quantity. The rate at which the soluble fatty acids were dissolved out from the insoluble fatty acids was also an important factor, depending upon the extent of the exposed surface of the fatty acids. In the Polenske process the exposure of surface was carried as far as possible to the limiting value by the use of a considerable quantity of very finely divided pumice, in order to approximate to a constant condition. He could not help thinking that the authors would have found the solution of the problem easier had they treated it mathematically. Messrs. Revis and Bolton had approached the matter in an entirely different way, and their combination of the Kirschner and Polenske methods seemed likely to give good results in the determination of the proportion of butter-fat in margarine. Their experience in regard to the use of a jacketed still-head in the Polenske process was much the same as his own. The only advantage of the jacketed still-head was that, except where the quantity of insoluble volatile acids was very large, it allowed of a greater latitude in regard to the time of distillation. In the ordinary Polenske process the limitation as to time of distillation was somewhat inconvenient, but when a jacketed still-head was used, it made practically no difference, with ordinary butter, whether the distillation was carried out in exactly twenty minutes, or continued for half an hour or even longer. When, however, large proportions of cocoanut oil were present, it was necessary always to distil for the exact time laid down, because, unless the boiling was very vigorous, the fatty acids were not mixed up, and the same surface of the insoluble fatty acids was not exposed to the aqueous portion.

Mr. T. Macara thought that the Polenske process should be included in any standard process of butter and margarine analysis that might be laid down, and that the inclusion of Messrs. Bolton and Revis's modification of the Kirschner method also would be additionally useful, particularly in the case of margarines containing cocoanut and similar oils. He had tried Messrs. Revis and Bolton's modification of Shrewsbury and Knapp's process, and had found it to be a great improvement.

Mr. E. Hinks, referring to Messrs. Revis and Bolton's statement that the presence of as little as 2 per cent. of butter-fat could be detected by means of the Kirschner process, asked if this held good when larger proportions of butter were present—*i.e.*, would the difference between, say, 10 per cent. and 12 per cent. of butter-fat be shown?

Mr. Cribb said that Mr. Richards and he had felt that the data they had so far obtained were not sufficient to permit of their treating the subject mathematically. They had brought their work forward merely as an attempt to open the way for the adoption of the combined Reichert and Polenske processes as a routine method, believing that, if an official process were to be laid down, it would be of advantage to choose one which was already familiar. Among the points in the official Reichert-Wollny method with regard to which they thought that modification was desirable was that of the temperature at which the distillate was filtered. It was inconvenient in warm weather to have to reduce the temperature to 12° C. by special means, and from this point of view 15° C. would be more satisfactory; but, on the other hand, the results were not so good as with the lower temperature. While pumice had been used in all the experiments recorded in their present paper, he should very much like to see it replaced by aluminium wire, which was first suggested to him by Mr. Stokes. The slow stream of hydrogen which the aluminium wire gave off most effectually prevented bumping; and when the wire was removed the residual fatty acids were available for further examination. The only objection that they had found to the use of aluminium wire was that the figures for soluble and insoluble volatile acids were altered, and a fresh basis for calculation would have to be worked out. He had been much interested in Messrs. Revis and Bolton's remarks about the Kirschner process, which appeared to supplement the Polenske process to a valuable degree just at the point where it was weakest—namely, in the determination of the proportion of butter present.

Mr. Revis agreed that the use of pumice was open to some objection, but he was afraid that it could not be satisfactorily replaced. Aluminium wire was, of course, much more convenient, and was satisfactory in the Kirschner process, where there was no layer of fatty acids to be kept boiling; but, in order to get the full Polenske value, the fatty acids must be kept boiling, and with aluminium wire the boiling could not be so satisfactorily maintained as with pumice. In some comparison experiments which Mr. Bolton and he had made, using pure cocoanut oil, a Polenske value of only 11·2 was obtained when aluminium wire was used, whereas the same sample yielded a Polenske value of 16·1 when pumice was used. He was not sure whether the Kirschner process would be capable of distinguishing between 10 per cent. of butter-fat and 12 per cent., but in practice it would scarcely be necessary to do this, and in any case he considered that the Kirschner value would allow of a nearer approximation to the truth than could be obtained in any other way.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Examination of Bismuth Salicylate. H. Caron and D. Raquet. (*Ann. Chim. anal. appl.*, 1911, **16**, 177-179.)—The authors use the diphenylamine test for bismuth subnitrate in the following manner: A little of the sample is dissolved in warm dilute (1 : 10) hydrochloric or sulphuric acid, the cold liquid filtered from the crystallised salicylic acid, and about 1 c.c. of the filtrate added to 2 c.c. of a solution of diphenylamine sulphate in dilute sulphuric acid (1 : 10). On now pouring about 3 parts by volume of pure sulphuric acid down the side of the tube a blue ring will be produced in the presence of traces of the subnitrate. Another test is to add to a little of the sample 1 c.c. of pure sulphuric acid to decompose the salicylate, and then to dilute the liquid, and add an excess of ammonia solution. Any nitric acid liberated by the sulphuric acid will react with the salicylic acid to form nitro-derivatives which will give a yellow coloration with the ammonia. This test can be made quantitative. C. A. M.

Testing of Camphor. W. Lenz. (*Arch. Pharm.*, 1911, **249**, 286-298.)—The author has made a comparative examination of samples of camphor obtained from East Africa, together with a standard sample of Asiatic camphor and one of synthetic camphor. The melting-point is regarded by the author as a most valuable means for indicating the purity of a sample of camphor. Absolutely pure camphor melts at 178° C. The estimation of the specific rotatory power is not adapted to the valuation of crude camphor, because the latter contains impurities which possess a stronger dextrorotatory power than pure camphor. It is, however, a means for ascertaining whether the sample consists of natural dextrorotatory camphor. The estimation of the residue from the evaporation of the camphor on the water-bath affords a useful measure of its purity, but it requires a very considerable time. The conversion of the camphor into the oxime has been effected by an improved method of the author so as to yield 93 per cent. of the theoretical quantity, instead of 75-85 per cent., as formerly. In the author's experiments the yield of oxime appears to be proportional to the percentage of pure camphor in the sample, but the deficiency is too great to allow the method to serve for the accurate valuation of the camphor. The colour reaction with vanillin and hydrochloric acid can at the best only serve for the recognition of natural camphor. This coloration is not so characteristic as the red coloration produced by pure hydrochloric acid, containing 38 per cent. of real acid, with natural camphor. The quantity of residue insoluble in 10 parts of this strong acid is a good measure of the impurities of commercial camphor, and might serve as the basis of an accurate method. No single test is able to afford an accurate valuation of any sample of camphor; conclusions must be drawn from the general results of all the tests. J. F. B.

Quantitative Estimation of Cantharidine in Cantharides and in Tinctures. A. Kneip, N. Ney, and F. Reimers. (*Arch. Pharm.*, 1911, **249**, 259-285.)—This is a prize essay composed for the Deutscher Apotheker Verein, and constitutes a complete survey of the methods which have been proposed for the estimation of cantharidine in the various forms of the drug. The methods investigated are twenty in number, and, in conclusion, each of the authors recommends a different modification of one of the older methods. The various methods may be classified as follows: Extraction with solvents without any addition; extraction with solvents with the addition of alkalis—*e.g.*, ammonia, magnesia, sodium carbonate, sodium hydroxide; extraction with solvents with the addition of acids—(*a*) with benzene, (*b*) with chloroform. Ney recommends a modification of the Panchaud-Seigfried method, extraction with chloroform in presence of nitric acid, evaporating the chloroform slowly, and washing the residue with limited quantities of petroleum spirit, alcohol, and ether successively, the two latter solvents being previously saturated with cantharidine. Reimers adopts the methods of Fromme and the Ph. Ger. with modifications: extraction with chloroform in presence of hydrochloric acid, distillation of the chloroform, and extraction of the residue with petroleum spirit, collecting the remainder on a tared filter. The whole is subsequently washed with water containing a trace of ammonium carbonate and dried at 40° C., the weight being corrected for losses by solubility. The cantharidine so obtained is not quite pure, and may be purified by dissolving in acetone. The process recommended by Kneip has the advantage of yielding a perfectly pure cantharidine, melting at 214° C. Fifteen grms. of powdered cantharides are moistened with 3 c.c. of absolute alcohol containing 25 per cent. of dry hydrogen chloride, the alcohol is evaporated off at a low temperature, and the substance is extracted in a Soxhlet's apparatus with a mixture of 30 c.c. of petroleum spirit (b.-p. 50°-75° C.) and 50 c.c. of benzene for two hours. To the extract, after the evaporation of the solvent, are added 5 c.c. of a mixture of 10 parts of alcohol and 90 parts of petroleum spirit to dissolve the green fat, and the liquid is decanted off through a tared filter previously dried at 60° C. The crystals are washed with successive portions of 5 c.c. of the same solvent. The fat is also washed out of the filter in the same way, and the flask and filter are dried at 60° C. and again weighed. The free cantharidine in the sample may be estimated by extraction without previous acidification. The results are fairly satisfactory. According to Kneip, Spanish flies should yield a minimum of 0·65 per cent. of cantharidine by this method. This author also describes a simplified modification of his method for the estimation of cantharidine in tinctures. For the details of the manipulation the original paper should be consulted. J. F. B.

Microscopical Examination of Gambier. H. Brumwell. (*J. Soc. Chem. Ind.*, 1911, **30**, 475-477.)—Gambier is the evaporated extract of the leaves and twigs of the climbing shrub *Nauclea* or *Uncaria Gambir*. The insoluble matter amounts generally to 10 to 15 per cent., but by more careful manufacture it might easily be reduced to 3 or 4 per cent.; the tanning matter amounts to about 40 per cent., and soluble non-tannins to about 30 per cent. The insoluble matter consists mainly of leaf-débris and plant-hairs, the latter in great abundance. The cuticles of the leaf may be separated for microscopic examination by boiling successively with 5 per cent. sulphuric acid, 5 per cent. sodium hydroxide, nitric acid sp. gr. 1·2, and nitric acid with the addition of small quantities of potassium chlorate. The upper cuticle of the leaf exhibits very distinct cells, fairly regularly disposed and no hairs or stomata. In the lower cuticle the stomata are large, very distinct, in great profusion, and are devoid of hairs. Sago flour is occasionally used to adulterate gambier. In the analysis of gambier, which is apt to be of irregular composition, it is advisable to weigh out a large portion of the sample—say 300 to 500 grms.—and dissolve in water, boiling if necessary, and to make up the syrup to a known weight for analysis. J. F. B.

Analysis of Gum Arabic Pastilles. R. Malenfant. (*J. Pharm. Chim.*, 1911, **3**, 484-489.)—A sample of so-called gum pastilles which had the taste of a gum arabic product of good quality was found to have the following approximate composition: Dextrose, 25; sucrose, 25; gelatin, 35; and tapioca, 15 per cent. It also contained a small quantity of calcium sulphate, but not a trace of gum arabic.

C. A. M.

Distribution of the Phosphoric Acid in Milk. F. Bordas and F. Touplain.

(*Ann. des Falsifications*, 1911, **4**, 229-231.)—Estimations of the total phosphoric acid in the ash, in the lactoserum separated after coagulation of the milk by trichloracetic acid, and in the coagulum (fat and casein) after oxidation with nitric acid and potassium permanganate, gave the following results: In total ash, 1·663 grms.; in lactoserum, 1·279 grms.; and in coagulum, 0·319 grm. per litre.

C. A. M.

Composition of the Seeds of Leguminous Plants grown in Togo and German East Africa. C. Grimme.

(*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 547-553.)—The seeds of the different plants in the following table are largely grown as food products in Togo and German East Africa:

Name.		Water.	Proteins.	Fat.	N-Free Extractives.	Crude Fibre.	Ash.
		Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Cajanus indicus	Pea bean	11·23	20·00	1·22	58·78	5·38	3·39
Phaseolus Mungo	Mungo bean	10·10	23·15	0·79	57·75	4·48	3·85
,, *vulgaris* ...	Bean	13·23	18·62	1·69	58·34	3·01	5·11
,, *lunatus* ...	Moon bean	13·70	21·13	0·91	57·06	3·61	3·59
,, var. *albomaculatus*	Moon bean	12·92	16·71	1·00	63·16	3·20	3·01
,, *inamœnus* ...	—	12·98	22·58	1·26	55·69	4·05	3·44
Dolichos Lablab ...	Lablab bean	10·20	25·66	0·78	53·95	5·87	3·54
Vigna Catjang (14 varieties)	Vigna bean	11·31 to 12·63	17·80 to 28·05	0·86 to 4·33	53·35 to 65·13	2·13 to 4·72	2·89 to 3·69
Canavalia ensiformis ...	Fetish bean	12·95	26·43	2·81	48·92	6·64	2·25
Voandzeia subterranea	Angola pea (5 varieties)	10·94 to 11·84	17·07 to 24·70	5·70 to 6·44	49·44 to 57·60	5·39 to 7·17	2·52 to 2·80

C. A. M.

Cryoscopic Examination of Milk. L. Stœcklin.

(*Ann. des Falsifications*, 1911, **4**, 232-257.)—From the examination of about 2,500 samples of milk during four years the author concludes that a cryoscopic examination will distinguish between genuine and watered milk, notwithstanding differences of age or the addition of potassium bichromate to the former. The production of 1 grm. of lactic acid per litre increases the observed freezing-point of a milk from about 0·045° to 0·050° C., and the latter value may be accepted as the correction to be made in practice. In like manner 1 grm. of potassium bichromate increases the acidity by an amount equivalent to 0·614 grm. of lactic acid. The amount of lactic acid capable of influencing the PG in a bichromated milk may be found by means of the formula—

$$L = A - (1{\cdot}20 + Cr \times 0{\cdot}061),$$

where L represents the true lactic acid in grms. per litre, A the total acidity of the milk expressed in terms of lactic acid, and Cr the grms. of potassium bichromate per litre. The factor 1·20 for the "non-lactic acidity" is reduced to 1·08 in a milk containing 10 per cent. of water, to 0·96 for 20 per cent. of water, etc. The average of the estimations showed that 1 grm. of potassium bichromate increased the original freezing-point of the milk (PGI) by 0·018° C. The application of these calculations may be illustrated by the following results obtained with a milk containing 1·1 grms. of potassium bichromate per litre and 3·5 grms. of total acids expressed as lactic acid:

$$\text{Observed freezing-point} = -0{\cdot}490^\circ \text{ C.}$$

$$\text{True lactic acid, } 3{\cdot}5 - (1{\cdot}20 + 1{\cdot}1 \times 0{\cdot}61) = 1{\cdot}73.$$

$$\begin{array}{lr} \text{Correction for lactic acid, } 1{\cdot}73 \times 0{\cdot}050 = & -0{\cdot}0865^\circ \text{ C.} \\ \text{,, \quad ,, \quad bichromate, } 1{\cdot}1 \times 0{\cdot}018 = & -0{\cdot}0198^\circ \text{ C.} \\ \hline & 0{\cdot}106^\circ \text{ C.} \end{array}$$

$$\text{PGI} = 0{\cdot}490^\circ \text{ C.} - 0{\cdot}106^\circ \text{ C.} = 0{\cdot}384^\circ \text{ C.}$$

$$\text{Amount of water added to milk} = 100 - \frac{0{\cdot}384}{0{\cdot}550} \times 100 = 30 \text{ per cent.}$$

Whenever the PGI is lower than −0·530° C., the addition of water is certain, whereas in the case of a milk showing a PGI of 0·550 watering cannot be regarded as proved, whatever be the results of chemical analysis. The pathological occurrence of an excess of water in milk (physiological water) will show a normal PG (about 0·550° C.) accompanying chemical evidence of the addition of water. C. A. M.

Comparison of the Reductase Test with Other Hygienic Tests for Milk. C. Barthel. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 513-534.)—The author has made a comparative study of the results obtained with his reductase test (Analyst, 1908, **33**, 237) and with other hygienic tests for milk. In Auzinger's modification of the alcohol test (*Milchwirtsch. Zentralbl.*, 1909, **5**, 293) a definite quantity (5 c.c.) of the milk is shaken with an equal volume of 60 per cent. (by weight) alcohol, and a note taken whether any precipitation of casein occurs. If there is no trace of a precipitate the milk is fresh, whilst a fine flocculent precipitate is obtained with slightly sour milk and a coarse one with distinctly sour milk. The author's experiments with this test show that milk with an acidity of 21° (Thörner) begins to give the reaction, whilst in most cases milk with a lower degree of acidity gives a negative result. In three cases, however, a positive result was obtained in spite of the acidity being normal. One of these samples showed a reduction period of more than twelve hours, and contained 90,000 bacteria per c.c., and was thus satisfactory from the hygienic point of view. The second was a milk with a putrid odour, having a reduction period of eight minutes, catalase = 7·5 c.c., and bacteria 28,530,000 per c.c.; whilst the third was free from objectionable odour or taste, but had a reduction period of ten minutes, catalase = 6·6 c.c., and contained 12,090,000 bacteria per c.c. In the author's opinion, therefore, the alcohol test affords a sharp means of detecting incipient souring of the milk, but its hygienic significance is limited, and it affords no more information than the acid value. As regards the catalase test (Analyst, 1910, **35**, 256, 526), it was found that out of forty-eight samples of milk which fell within the normal limits for the test (0 to 2·5 c.c. of liberated oxygen), four contained more than 10,000,000 bacteria per c.c. When examined by the reductase method, however, these four samples all gave a shorter period of reduction than the normal period. Out of 102 samples examined, there were twenty in which there appeared to be no relationship between the number of bacteria and the catalytic activity. The experiments showed that 2·5 c.c. of oxygen cannot be accepted as the highest permissible limit for good milk, since samples of good quality relatively poor in bacteria gave results as high as 4·5 c.c. A catalase value above this limit, however, corresponded, as a rule, to bacteria in excess of 10,000,000 per c.c., and a very short decolorisation period in the reductase test. The chief value of the catalase test is to detect abnormal milk or that from diseased individual cows in a herd. In the reductase test a period of reduction of less than one hour corresponds to about 10,000,000 or more bacteria per c.c. In the case of milks containing 4,000,000 to 10,000,000 of bacteria per c.c., reduction took place within an hour in half the samples, whereas with a still smaller bacterial content the decolorisation period was almost invariably longer than three hours. Only in the case of one sample containing over 10,000 bacteria per c.c. did the decolorisation time exceed three hours. The number 10,000,000 may therefore be regarded as the extreme limit for the number of bacteria that may be estimated by the reductase test. Below that figure the number of bacteria does not stand in any direct relationship to the time of decolorisation. In milks containing 10,000,000 to 20,000,000 of bacteria per c.c. the decolorisation time varied from seven to thirty-five minutes, whilst in those containing over 20,000,000 it was nearly always less than fifteen minutes.

C. A. M.

Applicability of Recent Methods for the Examination of Milk. E. Philippe. (*Mitt. Lebensmittelunters. u. Hyg.*, 1911, **2**, 1-36; through *Chem. Zentralbl.*, 1911, I., 1447-1448.)—The author considers that a leucocyte count affords a valuable means of diagnosing disease of the udder (*cf.* Hewlett and others, ANALYST, 1910, **35**, 23), and that a high catalase number is a certain indication of disease (*cf.* Revis, ANALYST, 1910, **35**, 359). He agrees that no lower limit can be placed on this number, which he determines at 25° C. and calculates as c.c. of oxygen split off by 100 c.c. of milk. Of 200 samples tested in this manner, 114 were found to have a catalase number of less than 20, that of 51 lay between 20 and 30, that of 17 between 30 and 40, whilst 16 were found to have a catalase number exceeding 40.

The fermentation-reductase test, or M-reductase test of Sames (*Milchwirtschaftl. Zentralbl.*, 1910, **6**, 462), using methylene-blue solution free from formaldehyde, is said to be a certain means of determining whether milk is fresh or not; the reduction of the colour to its leuco-base is due to bacterial action. A milk which is not decolorised within three hours may be passed as normal. The reductase test, or M. reductase test of Sames (*loc. cit.*), in which methylene-blue solution containing formaldehyde is used, is of doubtful value (*cf.* also Römer and Sames, ANALYST, 1910, **35**, 407), but it is said that a normal milk should decolorise the reagent within ten minutes. The author attaches importance to the determination in milk samples of sediment, which should be effected by a gravimetric method. G. C. J.

Soluble Basic Constituents of the Mushroom. F. Kutscher. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 535-540.)—A sample of mushroom extract sold under the name of "hercynia" as a medicinal stimulant, was found to contain adenin, but no guanin or xanthin. There were also isolated arginin and a new base, $C_{19}H_{15}N_3O_2$, which could be regarded as histidin that had taken up three methyl groups. The hydrochloride of this base gave a strong diazo reaction and a white precipitate, but no coloration with Millon's reagent. With potassium mercuric iodide it yielded a copious precipitate soluble in excess; with iodine in potassium iodide solution a brown precipitate, and with saturated alcoholic mercuric chloride solution a crystalline white precipitate; its platinichloride separated in short transparent yellowish-red needles. Its aurichloride melted at 180° to 182° C. The mushroom extract also contained betain and cholin. Since the quantities of these bases and of the potassium present were insufficient to exert their specific activity, the author concludes that the good results obtained in the treatment of invalids with this extract must be attributed to the presence of substances not precipitated by phosphotungstic acid.

C. A. M.

Accurate Estimation of Nicotine in Tobacco and in Green Plants of Nicotiana Tabacum. R. Mellet. (*Schweiz. Wochensch. Chem. u. Pharm.*, 1911, **49**, 117-120; through *Chem. Zentralbl.*, 1911, I., 1561.)—Toth's process (ANALYST, 1902, **27**, 12) is very useful for the estimation of nicotine in solutions or extracts, but some further modification is required for its estimation in solid materials. According to the method now described, the finely cut material, 30 grms. in the case of dry tobacco and 250 grms. in the case of the green plant, is treated with boiling water in a well-closed flask, and after twenty-four hours the liquid is cooled and milk of lime added. The flask is again closed and allowed to stand for a further twenty-four hours with frequent agitation. The liberated nicotine is distilled off in a current of steam, in such a way that the volume of liquid in the flask is reduced. When the volume of distillate attains two to three times that of the original liquid the operation is stopped and the distillate is acidified with sulphuric acid. It is then evaporated to a small bulk with exclusion of air, and, after the addition of potassium hydroxide, the nicotine is extracted with ether. The ethereal solution, containing some ammonia, is concentrated until all the latter is eliminated. The remainder of the ether is evaporated at the ordinary temperature; the residue is dissolved in water and titrated with $\frac{N}{10}$ sulphuric acid. Estimations effected with known quantities of nicotine indicated that the total losses of the alkaloid amounted to 0·06 grm., which figure must be added to the values found. The accuracy of the process, taking the above correction into account, depends to a certain extent on the quantity of nicotine found—*e.g.*, about 0·5 grm.—and the error, calculated on the nicotine, is less than 1·5 per cent.; when calculated on dry tobacco it is, at the most, a few mgrms., and on green tobacco a few tenths of a mgrm.

J. F. B.

Estimation of Nicotine in Tobacco Extracts. W. Koenig. (*Chem. Zeit.*, 1911, **35**, 521-522.)—The author has obtained satisfactory results by Toth's method (ANALYST, 1902, **27**, 12), working in the following way: 6 grms. of the extract are treated in a dish with 10 c.c. of 20 per cent. sodium hydroxide solution, and then mixed gradually with sufficient gypsum to give a nearly dry powder. The mixture is shaken in a cylinder with 100 c.c. of a mixture of ether and light petroleum spirit; 25 c.c. of the clear solution are transferred to a flask with 50 c.c. of water and an excess of $\frac{N}{10}$ hydrochloric acid, and then titrated back with $\frac{N}{10}$ sodium hydroxide in presence of iodeosin as indicator. The method suffers, however, from the defect that the results include any other bases which may be present in addition to nicotine. The author has introduced a polarimetric modification of this method by substituting a less volatile solvent—*e.g.*, toluene—for the ether-petroleum mixture. As a preliminary he has determined the specific rotation of pure nicotine in toluene solution, which may be taken as $[\alpha]_D = -168°$—that is, 1 grm. of nicotine dissolved in toluene to 100 c.c. gives a rotation in the 2 dcm. tube of $-3{\cdot}36°$ at 20° C. The modified procedure is as follows: 20 grms. of tobacco extract are treated in a porcelain dish with ignited sea-sand and 4 c.c. of sodium hydroxide (1 : 1) rubbed down to a semi-dry mass, and then mixed with sufficient gypsum to form a free powder. This is ground in a mortar and transferred to a stoppered bottle, the mortar being wiped out with sand and gypsum; 100 c.c. of toluene are added to the mass, the bottle is well stoppered and shaken for one to three hours. After settling, 30 to 40 c.c. are filtered through a covered filter and polarised. The rotation in the 2 dcm. tube, divided by 3·36, gives the weight of nicotine in 100 c.c.; but, since nicotine dissolves in toluene without loss of volume, a correction must be applied, thus: $x = g.\ \frac{100+g}{100}$, where $g =$ the grms. of nicotine found per 100 c.c.; then x multiplied by 5 gives the percentage of nicotine in the extract. For the titration of the nicotine, 25 c.c. of the filtered solution are placed in a stoppered flask in which a measured excess of $\frac{N}{10}$ hydrochloric acid and 50 to 75 c.c. of water are present. After the addition of 25 c.c. of ether and a few drops of iodeosin, the liquid is shaken vigorously, and the excess of acid is titrated back. One c.c. of $\frac{N}{10}$ acid = 0·0162 grm. of nicotine; the result is corrected in the same manner as above. The results are concordant, and agree satisfactorily with those obtained by Kissling's method; the author considers that Ulex's method cannot give correct results. If the polarimetric method alone be relied on, the tobacco extract may be made alkaline and shaken out with toluene direct, but the presence of ammonia, in that case, precludes the confirmatory titration.

J. F. B.

Adulteration of Pepper Corns. L. Bussard and Andouard. (*Ann. des Falsifications*, 1911, **4**, 263-266.)—A sample of pepper examined by the authors was found to be adulterated with the seeds of a species of lentil (*Ervum ervilia*), the appearance of which had been disguised by maceration in pimento extract and subsequent slight roasting until they had turned black. The microscopical appearance of the starch cells was very characteristic, being irregular in form, either elliptical or kidney-shaped, and having concentric striations. They ranged from 35 to 60 μ in length and from 30 to 40 μ in breadth, and were thus about six times the size of pepper starch cells. The seeds separated from the sample gave the following analytical results: Starch, 41·60; nitrogenous substances, 25·00; cellulose, 14·70; mineral matter, 3·26; and alcoholic extract, 4·36 per cent. They thus differed from pepper in containing much more starch and nitrogenous substances, but much less cellulose and rather less ash. The alcoholic extract was greatly increased by the preliminary treatment of the seeds with the pimento extract, that from the normal seeds being very small. C. A. M.

Titration of "Salvarsan" with Iodine Solution. G. O. Gaebel. (*Arch. Pharm.*, 1911, **249**, 241-247.)—"Salvarsan"—*i.e.*, dihydroxydiaminoarsenobenzene hydrochloride—contains 31·6 per cent. of arsenic, and its formula may be written $C_{12}H_{12}N_2O_2As_2.2HCl + 2H_2O$. It is readily oxidised by iodine, giving the corresponding arsinic acid, and the end-point of the titration is sharp. Instead, however, of the theoretical 8 equivalents of iodine, only 7·5 equivalents are reduced per molecule of salvarsan, owing to the reversibility of the reaction. For the titration, a 0·2 to 0·8 per cent. solution of salvarsan is prepared, and a quantity corresponding to 0·1 grm. of substance is measured out. This is titrated with iodine, with or without the addition of starch. Towards the end-point the colour of the iodine disappears somewhat slowly, but there is no difficulty in recognising the point of permanent coloration. The results, when calculated by the empirical factor, 1 c.c. of $\frac{N}{10}$ iodine $= 0·006326$ grm. of salvarsan, are perfectly satisfactory. J. F. B.

Customs Examination of Rye and Wheat Bran. R. Woy. (*Zeitsch. öffentl. Chem.*, 1911, **17**, 101-109; through *Chem. Zentralbl.*, 1911, I., 1447.)—The classification of bran according to its ash content was irrational, and the more recent official German method, in which account is taken of the proportion which passes a fine sieve, is not much better. A starch estimation is the only rational basis of classification, and should be prescribed now that quick and sufficiently accurate methods are available. The author prefers the method of Ewers (*Zeitsch. öffentl. Chem.*, 1909, **15**, 8) to that of Lintner (*cf.* Thorne and Jeffers, Analyst, 1909, **34**, 332), because it avoids the use of concentrated acid. Using Ewers' method with 5 grm. substance, and polarising in a 200 mm. tube at 20° C., the author finds the correct factor (assuming the use of the Soleil-Ventzke scale) to be 1·892. It is suggested that some substance other than coal-dust might be prescribed for denaturing bran—some substance that would not detract so much from its commercial value, whilst attaining the object of the Customs authorities. G. C. J.

Detection of Invertase in Wine. F. Schaffer. (*Mitt. Lebensmittelunters. u. Hyg.*, 1911, **2**, 36-40; through *Chem. Zentralbl.*, 1911, I., 1450.)—Müller-Thurgau (*Landw. Jahrbb.*, 1885, 795) was unable to detect invertase in finished clarified wines, and suggested that their capacity to hydrolyse cane-sugar, which had been alleged by earlier authors, must be due to their acidity alone. He worked on two portions of wine, one of which was heated nearly to 100° C. to destroy invertase, before they were each mixed with an equal bulk of 20 per cent. cane-sugar solution and incubated. The heated sample served as a control, and only if the other gave rise to detectably more reducing sugar under identical conditions did he admit the presence of invertase. The method is not sufficiently delicate for the detection of traces of invertase. The author of the present paper dialyses the wine to be tested for two or three days, at the end of which time the portion which cannot penetrate the membrane is neutral and free from any trace of reducing sugar. It is then mixed with a 5 per cent. solution of cane-sugar and incubated at 50° C. Tested in this way, every sample of wine experimented with, whether genuine or admittedly artificial, showed the presence of traces of invertase. G. C. J.

Sulphurous Acid in White Wines. P. C. Mestre. (*Ann. des Falsifications*, 1911, 4, 266-276.)—A complete analysis is given of 103 samples of white wine made in Gironde during several years since 1893, and of 34 commercial samples. In estimating the amount of free and combined sulphurous acid, a correction of 14 mgrms. of sulphur dioxide per litre was made in each case, this figure corresponding to the average amount of iodine consumed by the musts produced in the South-West of France. The average results of some of these years are summarised in the following table:

Wine.	Origin.	Total Sulphur Dioxide Mgrms. per Litre.			Free Sulphur Dioxide Mgrms. per Litre.		
		Max.	Min.	Mean.	Max.	Min.	Mean.
Wine in bottles of 1901, 1902, 1903, 1904, 1905	Rive gauche	288	230	259	53·90	31·40	40·13
	Rive droit	345	310	327	76·80	23·00	51·60
	Graves	257	194	225	34·00	16·50	25·25
Wine in casks of 1906, 1907, 1908, 1909	Rive gauche	349	149	239·70	77·00	16·97	43·38
	Rive droit	395	182·75	290·25	61·00	36·25	48·50
	Graves	215	95·00	192·75	81·90	14·80	43·47
	Entre 2 Mers	294·75	126·00	215·00	72·90	16·33	43·50
Typical commercial samples	Rive gauche	362	203	307	84·00	23·00	61·00
	Rive droit	450	212	361	93·00	38·00	64·00
	Graves	446	288	388	89·60	39·40	68·20
	Entre 2 Mers	370	238	300	113·90	6·40	34·70
Wine in casks of 1906, 1907, 1908 (excluding 1909)	Rive gauche	404·6	180·5	282	87·60	17·90	47·50
	Rive droit	420·0	210·3	320	63·33	40·60	50·60
	Graves	273·0	118·6	247	105·30	17·13	54·81
	Entre 2 Mers	408·5	101·6	260	76·00	22·00	33·00

C. A. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Fungicidal Action of Bordeaux Mixtures. B. T. P. Barker and C. T. Gimingham. (*J. Agric. Sci.*, 1911, **4**, 76-94.)—It is uncertain how the mixture of copper sulphate and lime known as Bordeaux mixture, containing no soluble copper, acts as an efficient fungicide, and the three views generally held as to how the copper present is rendered soluble are—(1) The chemical action of the CO_2 in the air or rain-water; (2) the solvent action of the leaves of the host plant; (3) the solvent action of the fungus itself. Gimingham (*J. Agric. Sci.*, 1911, 4, 69) and both authors in the present paper give reasons for not accepting the first view, and describe experiments in connection with the second which go to prove that when healthy leaves are placed in ordinary Bordeaux mixture—*i.e.*, made with equal weights of copper sulphate and lime, and containing an excess of lime—no copper was found in solution. In the case of lime-water, Bordeaux precipitate, which is the basic sulphate (10 CuO, SO_3), containing no excess lime, very small amounts of copper, varying from 0·00014 to 0·0005 per cent., were brought into solution by the leaves, but this in all probability is due to an excretion of solvent juices from very small injuries on the leaves, and not to the action of the normal leaf-surface. As regards the action of the fungus itself, the authors describe many experiments with hanging-drop cultures of conidia of *Nectria*, spores of *Penicillium glaucum*, uredo-spores of *Puccinia hieracia*, etc., and, in the case of the first named, consider it proved that actual contact with particles of the insoluble copper compound is sufficient to cause death, although growth can occur in the immediate vicinity, provided the organism does not actually touch the copper. A solvent action on the part of the fungus thus occurs, which is not powerful enough, normally, to produce serious toxic effect, except when direct contact occurs, and does not result in the bringing into solution of sufficient copper to exercise a general fungicidal action over the whole leaf. Failure to demonstrate the presence of soluble copper in liquids containing simply the basic sulphate or carbonate of copper and the spores, cannot be held to prove the absence of a solvent action on the part of the fungus. The question of distance between the copper particles and the fungus is the critical matter. All the results obtained by previous workers can be explained according to the authors' view, which is briefly as follows: The cell wall is to be regarded as the most important factor. In an isolated cell an interchange of material between the cell and the surrounding medium is always proceeding through the cell-wall, which normally is readily permeable by a variety of substances, although such interchange is probably reduced to a minimum in the case of thick-walled resting spores. The typical primary cell-wall is always more or less saturated with aqueous solutions, containing such of the cell products as are allowed to pass through the parietal protoplasmic layer, and these are capable of exerting a solvent action on insoluble particles of the copper compounds with which they come into actual contact, the toxic action of the copper then being diffused throughout the cell contents. The authors consider that both the toxic action of the spray on fungus already present, and an indirect protective action on the foliage rendering it more or less immune to subsequent infection, occur, and they discuss the work of Massee in this connection. The importance of a fine state of division of the copper precipitate and of *thorough* spraying is emphasised, attention being drawn to the inadvisability of having too great an excess of lime present, and to the advantages of using a spray with good adhesive qualities. H. F. E. H.

Influence of Metals in Presence of Hydrogen Peroxide on Blood Reagents. F. Michel. (*Chem. Zeit.*, 1911, **35**, 471.)—It is found that clean iron or copper in presence of hydrogen peroxide will oxidise guaiacum, Adler's reagent, leuco-malachite green, etc. Since metallic particles may be present on clothing, especially that of metal-workers, this observation has an important bearing on the detection of blood in such cases. If to the oxidised (coloured) solution a few c.c. of a 1 per cent. colourless solution of 2·4 diaminophenol be added, no immediate change will be observed if the original colour was induced by the catalytic action of blood or pus. Only very slowly does a red or dirty brown colour, due to oxidation of the diaminophenol by the air, appear. If the original colour was due to the action of iron or copper or their oxides, addition of diaminophenol gives rise to an immediate red coloration (which contrasts best with that of malachite green) of the three reagents named.

This method of application of the diaminophenol confirmatory test only serves, however, to distinguish organic oxidising catalysts from such inorganic or organic oxidants as are reoxidised by hydrogen peroxide. Chlorine or permanganate, for example, would develop the colour of malachite green and be destroyed in the process, and so behave like blood and give rise to no red colour on the subsequent addition of the diaminophenol. The substance is therefore first tested with leuco-malachite green without the addition of hydrogen peroxide. The development of a green colour indicates the presence of an oxidising agent proper, such as ferric oxide or chlorine. If no coloration is produced, hydrogen peroxide is added. Whether any colour then produced is due to metallic iron or copper, or to blood or pus, is determined by means of diaminophenol. If paper transfers have to be examined, these are dipped in solutions of the appropriate reagents. G. C. J.

Bacteriological Study of Honey. A. Sartory and E. Moreau. (*Ann. des Falsifications*, 1911, **4**, 259-263.)—Bacteriological examination of samples of honey from different parts of France detected the following species of bacteria within the cells of the comb: *B. subtilis*, *B. megaterium*, *Sarcina lutea*, *B. aerophilus*, *Micrococcus radiatus*, and *Staphylococcus pyogenes*. The mould isolated included the following: *Penicillium glaucum*, *Rhizopus nigricans*, *Mucor racemosus*, *Aspergillus gracilis*, *Sterigmatocystes nigra*, and yeast. Among the bacteria was a yellow species which liquefied gelatin, and appeared to be intermediate between the *B. luteus* of Flugge and the *B. flavus* of Macé. The yellow pigment secreted by this bacillus was soluble in absolute alcohol. It was produced most abundantly at the optimum temperature (30° C.) for the growth of the organism. C. A. M.

ORGANIC ANALYSIS.

Asphaltum from Mexican Petroleum. D. Lohmann. (*Chem. Rev. Fett- u. Harz-Ind.*, 1911, **18**, 107-108.)—A sample of asphaltum prepared from crude petroleum oil derived from two Mexican oil-fields was examined by the methods of Marcusson and Eickmann (ANALYST, 1911, 225). The original oil had a sp. gr. of 0·890 to 0·900 at 15° C., and yielded about 10 per cent. of petroleum spirit (benzine), 20 per cent. of lamp oil (kerosene), and 20 per cent. of asphaltum residue, together with lubricating oils, etc. The asphaltum gave the following results. Sp. gr. at

15° C., 1·07 to 1·12; total bitumen soluble in carbon bisulphide, 99·2 per cent.; soluble in normal benzine, 74 per cent.; volatile hydrocarbons, 76·5; carbon, 23·3; ash, 0·2; and sulphur, 5·5 per cent. Asphaltum of this type is exported in considerable quantities to Europe. Other samples contained from 2 to 6·2 per cent. of sulphur. Hence the proportion of sulphur affords no certain means of distinguishing between natural asphaltum and petroleum asphaltum. The proportion of ash in the Mexican petroleum products ranges from 0·1 to 0·9 per cent. There are also some kinds of natural asphaltum (*e.g.*, gilsonite) containing only 0·5 per cent. of ash. The amount of oil separated from the first sample by the method of Marcusson and Eickmann (*loc. cit.*) was 32·2 per cent. It was dark brown and of the consistence of cylinder oil, but was quite free from vaseline-like particles. It contained 2·2 per cent. of paraffin wax, estimated by the method of Holde. The acid value of the first distillate of 15 c.c. from 100 grms. of the asphaltum was 0·9 (after washing with water), whilst the second fraction of sp. gr. 0·8196, had an acid value of 0·42. The acid values of these distillates were therefore in accordance with the statement of Marcusson that the first fraction should have an acid value of less than one, and the second fraction a very small value. Both the value and the amount of paraffin wax, however, are so close to the limits given by Holde and Marcusson for petroleum asphaltum (paraffin wax, minimum, 2 per cent.; acid value, maximum, 1), that the latter should only be accepted with caution. C. A. M.

Wet Method for the Estimation of Carbon in Organic Substances. F. Tangl and G. v. Kereszty. (*Biochem. Zeitsch.*, 1911, **32**, 266-273.)—In the method described the organic substance is heated in a small flask with potassium bichromate and sulphuric acid until completely decomposed. The gases evolved are conducted from the flask through a combustion tube filled with copper oxide and heated to redness, and then through a tube filled with lead peroxide heated to a temperature of 150° to 180° C. From the latter tube the gases are conducted through an absorption vessel containing barium hydroxide solution, and, at the end of the experiment, the resulting barium carbonate is titrated in the usual way. A condenser is fitted in the neck of the decomposition flask, so that excess of moisture does not pass into the combustion tube, and the whole apparatus is so arranged that a current of dry air free from carbon dioxide may be passed through it before and after the organic substance has been decomposed; a specially-devised funnel fitted into the neck of the flask allows the substance and the sulphuric acid to be added after the current of pure air has been passed through the apparatus for some time. The method yields trustworthy results, even with substances containing nitrogen, sulphur, and chlorine. W. P. S.

Precipitation of Cocaine Solutions with Platinum Chloride. M. Nyman and R. Bjorksten. (*Pharm. Zentralbl.*, 1911, **52**, 71-74; through *Chem. Zentralbl.*, 1911, I., 1451.)—Cocaine, in not too dilute solution, may be estimated by precipitation with 2 c.c. of a solution of platinum chloride in hydrochloric acid (sp. gr. 1·125) followed by addition of 3 volumes of alcohol. As cocaine platinichloride is soluble to the extent of 0·012 per cent. in absolute alcohol, 0·022 per cent. in 70 per cent. alcohol, and 0·030 per cent. in 50 per cent. alcohol, a proper correction must be applied to the weight found. The method serves well for the control of sterilised cocaine solutions. The presence of boric acid has but little influence on the results, but the presence of sodium chloride renders precipitation incomplete, and the method is useless for the estimation of cocaine in presence of atropine. G. C. J.

Estimation of Free Fatty Acids in Fats. E. Bödtker. (*Chem. Zeit.*, 1911, **35**, 548.)—The author refers to Loebell's method for estimating the acid value of fats (*Chem. Zeit.*, 1911, **35**, 276). From 5 to 15 grms. of the fat (melted if required) are thoroughly shaken with 25 c.c. of alcohol, after which 50 c.c. of water and 2 to 3 drops of 1 per cent. phenolphthaleïn solution are added, and the mixture titrated with $\frac{N}{10}$ sodium hydroxide solution until the colour ceases to disappear when the flask is vigorously shaken, and then with $\frac{N}{10}$ hydrochloric acid until colourless. The method is advantageous in the case of coloured fats, since the dyestuffs are insoluble in water. C. A. M.

Estimation of Fatty Acids in Soaps. T. Budde. (*Verröffentl. a. d. Gebiete des Militärsanitätswesens*, Heft 5; *Arbb. a. d. hyg.-Chem. Unters.-Stellen*, 1911, **4**, 86-99; through *Chem. Zentralbl.*, 1911, I., 1449-1450.)—The soap is dissolved in absolute alcohol, the solution filtered from insoluble matter, any free alkali neutralised, and after neutralisation evaporated to dryness. The residue is dissolved in water and the solution made up to a known volume. Of this, 25 c.c. (corresponding to 0·5 to 0·8 grm. soap) is precipitated by the addition of 25 c.c. of a sufficiently strong solution of copper sulphate, and the mixture boiled. When cold, the copper salt of the fatty acids is filtered off, washed, dried at 103° C., and weighed. Assuming the copper salt to contain 10 per cent. of copper, its weight, less 9·7 per cent., should give the weight of fatty acids in the quantity of soap taken for precipitation. The results thus obtained are usually 1 per cent. above the truth, but never 2 per cent.

G. C. J.

Simplified Process for Estimation of Acid and Saponification Values in Wax. (*Pharm. Zentralbl.*, 1911, **52**, 363-367; through *Chem. Zentralbl.*, 1911, I., 1380.)—One grm. of the wax is heated for five minutes with 10 c.c. of absolute alcohol in a closed 100 c.c. bottle, which is suspended in a Nyman steriliser (*Chem. Zentralbl.*, 1910, I., 1402), and frequently agitated. After cooling for three minutes, the mixture is titrated with $\frac{N}{2}$ potash, using phenolphthaleïn as indicator; a further 20 c.c. of $\frac{N}{2}$ potash is then added, and the bottle closed, and heated for twenty-five minutes. It is then allowed to cool for three minutes, when the excess of potash is determined in the usual manner. G. C. J.

Estimation of Formic Acid. H. Franzen and F. Egger. (*J. prakt. Chem.*, 1911, **83**, 323-325; through *Chem. Zentralbl.*, 1911, I., 1529.)—The following improvement of the method of Franzen and Greve (ANALYST, 1909, **34**, 110; 1910, **35**, 29) is recommended: To each 0·5 grm. of the formic acid are added 50 c.c. of a clear solution of 200 grms. of mercuric chloride, 300 grms. of sodium acetate, and

80 grms. of sodium chloride, in a litre of water, and the mixture is heated for three and a half hours on the water-bath; the separated mercurous chloride is then estimated as described in the previous paper. It is essential that the concentration of the formic acid should not exceed 0·5 grm. per litre of liquid. C. A. M.

Detection of Graphite in Lubricants. J. Marcusson and G. Meyerhelm. (*Chem. Zeit.*, 1911, **35**, 461-462.)—The graphite which is incorporated with lubricating greases may generally be separated by treatment with benzene. Some greases are mixtures of fat and lime soaps; in such cases a mixture of 90 parts of benzene and 10 of alcohol by volume is a good solvent both for oil and soap. The graphite may be filtered off together with the mineral impurities, which are mostly soluble in hot hydrochloric acid; the graphite is estimated by combustion. In the case of mixtures of graphite with tars and pitches, the separation is more difficult, since these materials frequently contain carbon and black insoluble hydrocarbons not easily distinguished from graphite, especially if mixed with talc or similar mineral matters. In such cases a separation of the graphite from the amorphous forms of carbon may generally be effected by fusion with sodium hydroxide: One grm. of the residue insoluble in benzene is added in small portions at a time to 15 grms. of fused sodium hydroxide in a nickel crucible, and heated for fifteen minutes over a burner, and for a few minutes longer over the blowpipe flame. The amorphous carbon is oxidised by this treatment more rapidly than graphite. The melt is dissolved in water, the insoluble matter is washed by decantation, then on a filter; it is treated with hot dilute hydrochloric acid to remove iron and nickel hydroxides, and is finally dried at 105° C. Different varieties of graphite possess different degrees of resistance to oxidation, so the method is not absolutely reliable. The authors have been able to detect the presence of 20 per cent. of graphite mixed with the insoluble carbonaceous matters of pitches. In practice, however, the proportion of graphite, if present in sufficient quantity to have any appreciable lubricating value, will be considerably more than 20 per cent. of the insoluble carbon of the pitch. J. F. B.

Gum from Bombax Malabricum. P. P. Phillips. (*J. Soc. Chem. Ind.*, 1911, **30**, 469-471.)—The *Bombax malabricum* (vern., *semul*), the red, silk-cotton tree order *Malvaceæ*, yields a gum which exudes only from those portions of the bark which have been injured by decay or the attacks of insects (chiefly termites), but not from incisions in the healthy bark. The gum, known as *mocherus*, is sold in the form of brittle, mahogany-coloured tears, hollow in the centre. When powdered and treated with water, it swells up and yields a reddish-brown liquor, from which alcohol precipitates a little gum; the main bulk of the material is, however, insoluble in water. The gum leaves on incineration about 8 to 9 per cent. of ash. An extract obtained by boiling the gum for four hours with water gave many of the characteristic reactions of tannin. It gives a green coloration with ferric chloride, changing to purple on the addition of very dilute ammonia. It precipitates gelatin, and bromine water produces a copious yellow precipitate. The powdered gum dissolves almost completely in solutions of caustic alkalis. From its reactions, it is evident that the gum contains a considerable proportion of tannin, having the properties of a catechol tannin. On dry distillation of the gum, catechol could be detected in the distillate. During distillation, a pinewood shaving, moistened with hydrochloric acid and placed near the mouth of the receiver, was instantly coloured violet. On hydrolysis of the gum with boiling dilute hydrochloric acid, a red-coloured solution is obtained, with an insoluble residue having the colour of crimson lake. This colouring matter is only moderately soluble in alcohol; it does not possess the solubility generally attributed to the phlobaphenes. After removal of the tannins by lead acetate, the hydrolysed extract reduces Fehling's solution.

J. F. B.

Hydrargyrometric Analyses. L. Rosenthaler. (*Arch. Pharm.*, 1911, **249**, 253-259.)—Since mercuric nitrate gives a large number of reactions which yield undissociated or insoluble mercury compounds, it may be employed for volumetric analyses in many cases in preference to silver nitrate, using Volhard's method. The solutions required are: $\frac{N}{10}$ mercuric nitrate, $\frac{N}{10}$ ammonium thiocyanate, ferric ammonium sulphate, and concentrated nitric acid free from nitrous acid. An excess of mercuric nitrate is added to the liquid to be titrated, the mixture is shaken, and 2 c.c. of saturated ferric ammonium sulphate solution are added, together with sufficient nitric acid to produce perfect decolorisation. The liquid is then titrated back with thiocyanate until the red colour is established. The changes are not quite so sharp as in titrations of silver nitrate, but may be made sharper by saturating with sodium or potassium nitrate free from chlorides. Good results are obtainable in the estimation of hydrocyanic acid and cyanides, bromides, iodides (*e.g.*, ferric iodide in pharmaceutical preparations). The method is available for the estimation of hydrocyanic acid in presence of benzaldehydecyanhydrin, but not for the estimation of the combined hydrocyanic acid in the latter; it also cannot be used for the estimation of chlorides, since mercuric chloride is dissociated to a considerable extent in solution. J. F. B.

Oil from Seeds of the Ash-Tree. W. Bach. (*Chem. Zeit.*, 1911, **35**, 478.)—The crushed seeds of the ash-tree yielded, on extraction with ether, 9·7 per cent. of a brownish-yellow thick oil with an odour recalling that of tea. It formed a semi-solid mass in the elaidin reaction, and in its drying properties resembled sunflower and soya bean oils. A film exposed on glass at the ordinary temperature was not completely dry after fourteen days. The filtered oil became turbid at 27·1° C., and gave the following values: Sp. gr., 0·9181; saponification value, 168·5; iodine value, 129·5; Reichert-Meissl value, 1·68; insoluble fatty acids, 85·1 per cent.; unsaponifiable matter, 5·5 per cent. *Fatty acids:* Neutralisation value, 181·7; iodine value, 125·8; melting-point, 36·8° C.; and solidification-point, 28·6. The fresh oil contained 1·71 per cent. of free fatty acids (calculated as oleic acid). C. A. M.

Rice Oil. M. Tsujimoto. (*Chem. Rev. Fett- u. Harz-Ind.*, 1911, **18**, 111-112.)—A sample of commercial rice oil, obtained by extracting the crushed rice with light petroleum, was of a greenish-yellow colour, and had the following characteristics: Sp. gr. at 15° C., 0·9273; acid value, 34·75; saponification value, 184·87;

iodine value (Wij's), 107·6; $[n]_{D20^{\circ} C.}$, 1·4742. *Fatty acids:* Sp. gr. at 100° C. 0·8528 (without unsaponifiable matter, 0·8523); melting-point 30·5° C. (without unsaponifiable matter, 32·5° C.); neutralisation value, 183·72 (and 197·66); and iodine value, 109·47 (and 107·82). The unsaponifiable matter (4·78 per cent.) yielded a phytosterol melting at 136° to 137° C. The fatty acids, examined by the usual method, were found to consist of approximately 20 per cent. of palmitic acid, 45 per cent. of oleic acid, and 35 per cent. of an isolinolic acid which yielded a sativic acid melting at 158·5° to 159° C., instead of the usual 173° to 174° C. The mixed fatty acids gave no insoluble bromide. C. A. M.

Niger-Seed Oil. K. Utz. (*Chem. Rev. Fett-u. Harz.-Ind.*, 1911, **18**, 106-107.) —Samples of crude and bleached niger-seed oil gave the following analytical values:

NIGER-SEED OILS.

	Sp. Gr. at 15° C.	Acid Value.	Saponification Value.	Iodine Value.	Reichert-Meissl Value.	Refraction $[n]_{D15^{\circ}}$ C.	Unsaponifiable Matter. Per Cent.
Crude oil ...	0·9252	3·70	123·5	126·8	0·88	1·4768	1·26
Bleached oil ...	0·9250	0·45	217·8	114·0	3·85	1·4764	0·53

FATTY ACIDS.

	Melting-Point, ° C.	Iodine Value.	Refraction $[n]_{D40^{\circ}}$ C.
Crude oil	28·2	113·8	1·4605
Bleached oil	27·8	102·3	1·4605

The sample dried very slowly, even at 100° C., and in this respect did not agree with Allen's statement that niger-seed oil dried rapidly at that temperature.

C. A. M.

Soya Bean Oil. C. Oettinger and F. Buchta. (*Zeitsch. angew. Chem.*, 1911, **24**, 828-829).—Samples of crude and refined soya bean oil gave the following results on analysis. The figures for the saponification and iodine values and melting-points of the fatty acids differ widely from those previously published. The solidification-point (+8° to +16° C.) given by De Negri and Fabris, and copied in many textbooks, is evidently due to a clerical error:

OILS.

Soya Bean Oil.	Solidification-Point.	Sp. Gr. at 15° C.	Acid Value.	Saponification Value.	Iodine Value.	Hehner Value.	Reichert-Meissl Value.	Refractive Index at 20° C.	Unsaponifiable Matter.
	° C.								Per Cent.
Finest ...	− 8 to − 16	0·9255	0·0	192·9	135·0	95·95	0·45	1·4755	0·59
Common ..	− 8 to − 16	0·9246	0·41	192·4	132·9	96·01	0·50	1·4750	0·54
Crude ...	− 8 to − 16	0·9265	5·2	194·0	135·0	95·85	0·56	1·4755	0·39

FATTY ACIDS.

	Melting-Point.	Solidification-Point.	Refractive Index at 27·5° C.
	° C.	° C.	
Finest	26	22	1·465
Common	26	22	1·465
Crude	26	22	1·465

C. A. M.

Detection of Train Oil in Oils, Fats, and Soaps. J. Marcusson and H. v. Huber. (*Seifensieder-Zeit.*, 1911, **38**, 249-251; through *Chem. Zentralbl.*, 1911, I., 1448.)—The following simplified modification of Lewkowitsch's method is recommended: The separated fatty acids (10 grms.) are shaken in a stoppered cylinder with 200 c.c. of a bromine solution (1 part bromine, 28 parts acetic acid, 4 parts nitrobenzene). If no precipitate appears after standing an hour, the sample is free from train oil and from drying vegetable oils. If a precipitate appears, the mixture is allowed to stand another hour, and then filtered, using a small filter-plate and paper. The precipitate is washed on the filter with ether, weighed, and reduced to powder. It is then heated for half an hour under a reflux condenser with 50 times its weight of benzene, which dissolves the hexabromide of linolenic acid. Complete solution shows absence of train oil in the sample; but if anything remains undissolved, it is filtered off with the use of a hot-water jacketed filter, dried, and its melting-point determined. If it blackens, but remains unmelted at 200° C., it may be taken to be the octobromide characteristic of the train-oil group. If it melts lower than this (hexabromolinolenic acid melts at 175° to 180° C. without decomposition), it is re-extracted with hot benzene, and its melting-point re-determined. If, instead of soaps, oils have to be examined, these may be treated direct with Halphen's bromine solution. If no precipitate forms, train oil is absent; but if one forms, the experiment must be repeated on the separated fatty acids, as no convenient solvent is known for separating the brominated glycerides of train oil from those of vegetable drying oils. G. C. J.

Causes of Loss of Phosphoric Acid on Incineration in Presence of Organic Matter. E. Fleurent and L. Levi. (*Bull. Soc. Chem.*, 1911 [iv.], **9**, 379-393.)—There are at least three causes of loss of phosphoric acid when organic matter containing it is burnt. Of these, the reduction of a portion of any acid phosphates present and combustion of the resulting phosphorus with escape of phosphorus pentoxide has long been recognised and met to some extent by addition of alkali. Addition of sufficient alkali also tends to negative the second cause of loss—namely, the expulsion of phosphoric acid from its compounds by silica; but the addition of solid caustic lime, as is sometimes practised, has but little effect in preventing either of these causes of loss, as it cannot be brought into sufficiently intimate contact with the substance undergoing incineration. A third cause of loss is in some way connected with the fatty constituents of the organic matter, and in some animal matter the loss due to this cause alone may exceed 25 per cent.

So far as the estimation of total phosphorus is concerned, all the above difficulties may be surmounted by destruction of organic matter in the wet way, either by means of sulphuric acid alone, or, preferably, by the successive action of fuming nitric acid and sulphuric acid. But if it be desired to make an exhaustive analysis of the ash of any organic body, incineration becomes necessary, and the authors have worked out the following method: The organic matter is first freed from fat or oil by extraction with ether. It is then carbonised at a low temperature in a closed crucible. The carbonised mass is pulverised, returned to the crucible, and moistened with a known quantity of lime-water, milk of lime, or baryta solution. Working on 10 grms. of substance, it suffices to add 0·05 grm. lime for each 1 per cent. of phosphoric anhydride possibly present. After evaporating to dryness, the contents of the crucible are transferred to one or more boats, which are placed in a combustion-tube in a furnace, and raised to a low red heat whilst a current of carbon dioxide is passed through the tube. The stream of carbon dioxide is then replaced by a slow one of oxygen, when a white ash containing the whole of the phosphoric acid and chlorine of the original organic matter is obtained. When combustion is complete, the temperature is raised for a few minutes to decompose any calcium carbonate. In estimating the total ash or its lime content, account must, of course, be taken of the weighed quantity of lime used in the process. G. C. J.

Colorimetric Method for the Estimation of the Molecular Weight of Polysaccharides. L. Wacker. (*Zeitschr. f. physiol. Chem.*, 1911, **71**, 143-152; through *Chem. Zentralbl.*, 1911, I., 1380-1381.)—The principle of the method has been described in earlier papers (ANALYST, 1908, **33**, 131; 1909, **34**, 410). It is now found that it is unnecessary to have recourse to the inversion method, so long as substances of the general formula $(C_6H_{10}O_5)_nH_2O$ are being investigated. The colour intensity is not exactly proportional to the number of carbohydrate residues present, but the error involved by assuming it to be so is less than 2 per cent., when the comparison is made between polysaccharides of 4 and 10 hexose residues, and this error is negligible in a determination of molecular weight. Lævulose and polysaccharides yielding lævulose on hydrolysis are exceptional, and give rise to colours of greater intensity than are required by theory. The latest directions for carrying out the test, which differ notably from those given earlier (ANALYST, 1908, **33**, 131), are as follows: To each of sixteen cylinders of equal diameter (about 30 mm.) a measured quantity of a 0·01 per cent. solution of a carbohydrate of known complexity (say lactose) is added, the quantities varying from 0·5 c.c. to 8 c.c. Enough water is added to bring the contents of each cylinder up to 10 c.c., and then 2 c.c. of 2 per cent. *p*-phenylhydrazine sulphonic acid and 2 c.c. of caustic soda solution (1:2) are added. In another cylinder 2·5 c.c. of a 0·01 per cent. solution of the carbohydrate to be tested are treated in a similar manner and the colour comparison made at the expiration of four hours, during which the contents of the cylinders are frequently agitated. G. C. J.

Polarimetric Estimation of Sugar by the Method of Jolles. B. Bardach and S. Silberstein. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 540-543.)—If air be admitted to the vessel during the heating of the sugar with the alkali in a thermostat, much less discoloration occurs, and it is therefore possible to employ more concentrated solutions and to polarise the liquid in a 200 mm. tube without dilution or treatment with lead acetate, etc. Experiments with solutions of dextrose (0·5 to 5 per cent.) showed that after twenty hours' heating there was invariably lævorotation, so that in applying the method an empirical value of $-0{\cdot}25°$ is taken as the end of the reaction. A measured quantity of the sugar solution under examination is mixed with sufficient $\frac{N}{1}$ sodium hydroxide solution to render the alkalinity of the liquid decinormal. The solution is polarised, and an aliquot portion placed in a beaker 8 to 10 cm. high by about 5 cm. in diameter, and heated uninterruptedly for twenty hours in a thermostat maintained at about 37·5° C. (36° to 39° C.). The liquid is then cooled, made up to its original volume and again polarised. The proportion of dextrose (D) in mixtures of dextrose and sucrose may be found by means of the formula $D = A - B - 0{\cdot}25$—and that of the sucrose (S) by the formula $S = B + 0{\cdot}25$, where A represents the polariscope reading before, and B that after the action of the alkali. The results tabulated show that the method is accurate (*cf.* ANALYST, 1911, 28). C. A. M.

Estimation of Rubber as Tetrabromide. Decomposition of the Bromide by Nitric Acid. D. Spence, J. C. Galletly, and J. H. Scott. (*Gummi-Zeit.*, 1911, **25**, 801-803; through *Chem. Zentralbl.*, 1911, I., 1451-1452.)—When caoutchouc tetrabromide is decomposed by nitric acid, some bromine is lost, even when silver nitrate is present in excess, and when the initial concentration of nitric acid is kept lower than is specified by Budde in describing his process. The loss of bromine increases with increasing acid concentration, and appears to depend in part on factors which cannot be controlled. Working with much weaker acid than Budde recommends, the authors find that the unavoidable loss of bromine and corresponding error in the caoutchouc determination is about 2 per cent. Following Budde's directions more closely, the results are even lower. G. C. J.

INORGANIC ANALYSIS.

Titration of Alkali Carbonates in Presence of Alkali Hydroxides or Bicarbonates. J. Tillmanns and O. Henblein. (*Zeitsch. angew. Chem.*, 1911, **24**, 874-876.)—It is shown that the separate estimation of alkali hydroxide and carbonate or of carbonate and bicarbonate by titration with acid in two stages, using phenolphthaleïn as indicator in the first stage and methyl orange in the second, may give rise to inaccurate results. Though there may be no visible escape of carbon dioxide during the first stage of the titration, variable amounts of carbon dioxide do, in fact, escape, and the titration should be conducted in stoppered flasks, the stoppers of which should be replaced and the contents shaken after each small addition of acid. In this way more accurate results may be obtained.

An alternative method which gives equally good results is to titrate one portion of the solution, using methyl orange as indicator. To a second and equal portion there is then added the volume of standard acid required in the methyl orange titration; but, instead of using methyl orange, phenolphthaleïn is added to this portion, and the carbon dioxide is titrated against standard caustic alkali, using a stoppered flask, as already described. G. C. J.

Microchemical Test for Aluminium by Means of Caesium Chloride. N. Schoorl. (*Zeitsch. anal. Chem.*, 1911, **50**, 266-267.)—Pure caesium chloride, when added to dilute solutions of aluminium in sulphuric acid, does not give rise to crystals of caesium alum. If, however, the caesium chloride be mixed with a minute proportion of finely powdered caesium alum (1 : 1,000,000), the formation of relatively large crystals of caesium alum is rapid. G. C. J.

Estimation of Arsenic in Pyrites. G. Hattensaur. (*Oesterr. Zeitsch. f. Berg.- u. Hüttenwesen*, 1911, **59**, 175-178; through *Chem. Zentralbl.*, 1911, I., 1378.)—12·5 grms. pyrites contained in a tall beaker are covered with 10 c.c. water and 1 c.c. sulphuric acid, and nitric acid is added gradually till frothing ceases. The mixture is evaporated to a syrupy consistency and then dissolved in boiling water. If the residue is other than white, the solution is decanted into a 250 c.c. flask and the residue is then treated with aqua regia. After evaporating the excess of acid, the solution and white residue are transferred to the 250 c.c. flask, which is filled to the mark, and the contents shaken and filtered. Into 200 c.c. of the filtrate (corresponding to 10 grms. pyrites) hydrogen sulphide is led until the precipitate coagulates, leaving the supernatant liquor clear. The sulphides of arsenic and copper (and antimony if present) are filtered off and the arsenic sulphide dissolved by digestion with ammonium carbonate. The filtered solution is evaporated on the water-bath after addition of some nitric acid. It is then diluted, an excess of silver nitrate added, and the solution exactly neutralised with ammonia, and warmed. The precipitate of silver arsenate is filtered off and washed with cold water till the washings are free from silver. Finally, the precipitate and filter are dried at the mouth of the muffle and cupelled with lead. The arsenic in the pyrites is then calculated from the weight of the silver, after correction for cupellation loss and for any silver in the assay lead.

G. C. J.

Estimation of Arsenic in Schweinfurt Green. A. Heiduschta and A. Reuss. (*Zeitsch. anal. Chem.*, 1911, **50**, 269.)—The sample (0·5 grm.) is mixed in a 150-200 c.c. round-bottomed flask with 5 grm. of ferrous sulphate in small crystals and 60 c.c. of fuming hydrochloric acid. The mixture is distilled, and, after passing up through a spraytrap, the vapours are led down into a 700 c.c. Erlenmeyer flask provided with a double-bored stopper, the second hole of the stopper carrying a Péligot-tube. Flask and Péligot-tube contain caustic potash solution, about 40 grms. of KOH in all, and the flask stands in ice-water. When three-quarters of the acid has been distilled, the apparatus is disconnected and the flame withdrawn. The cooled distillate is neutralised with hydrochloric acid, a considerable quantity of sodium bicarbonate is added, and the arsenic titrated with iodine as usual.

G. C. J.

Method of Measuring the Degree of Vitiation of the Atmosphere. H. Henriet and M. Bouyssy. (*Comptes rend.*, 1911, **152**, 1180-1181.)—Since the animal excretory products to which the vitiation of air is due are all reducing bodies, a measurement of the reducing capacity of the air affords an index of its degree of purity. These excretory products may all be condensed simultaneously with the moisture in the air, so that by condensing a given volume of moisture, calculating the corresponding volume of air, and estimating the reducing substances in the condensed water, comparative values are obtained. For the condensation, the authors use a conical nickel vessel holding about 3 litres, within which is fitted another vessel containing a mixture of ice and salt. From the volume of the liquid condensed upon the cylinder the corresponding volume of air is calculated by means of the formula:

$$v = \frac{m\ (1+\alpha t)}{ad}\ \frac{76}{f},$$

where v represents the volume of the mixture of air and water vapour at t° C. and at the pressure H; m, the weight of the vapour; a, the weight of 1 c.c. of air; d, the sp. gr. of the vapour, and f its tension. Reducing the volume of the mixture to 0° C. and a pressure of 760 mm.:

$$v_0 = \frac{1}{ad}\ \frac{m\mathrm{H}}{f} = 1{\cdot}243\ \frac{m\mathrm{H}}{f}.$$

For estimating the reducing substances, 5 grms. of the condensed water vapour are boiled for five minutes in a 50 c.c. flask with 10 c.c. of a reagent containing 316 mgrms. of potassium permanganate and 425 mgrms. of pure chromic anhydride per litre. The liquid is then cooled, treated with 10 c.c. of a solution containing per litre 10 grms. of ferrous ammonium sulphate and 10 c.c. of pure sulphuric acid (sp. gr. 66° Bé.), and titrated with $\frac{N}{500}$ potassium permanganate solution. A blank estimation is made without the condensed moisture, and the difference between the two results gives the amount of permanganate corresponding to the reducing substances derived from the air. The result is calculated into the number of mgrms. of oxygen per 100 cubic metres of air. In this way the following results were obtained: Atmospheric air (Montsouris Park), 1; air from vicinity of Hôtel de Ville, 10; from room containing animals (rabbits, monkeys, etc.), 13; printing works, 13; badly ventilated office, 14; badly ventilated passage, 17; and dressmakers' workroom, with windows shut, containing twenty workpeople, 21.

C. A. M.

Quantitative Separation of Barium, Strontium, and Calcium. J. L. M. van der Horn van den Bos. (*Chem. Weekblad*, 1911, **8**, 5-10; through *Chem. Zentralbl.*, 1911, I., 1379-1380.)—The diluted solution, acidified with acetic acid, is heated to boiling, and a slight excess of ammonium chromate solution added drop by drop, stirring continuously. When cool, the liquid is decanted through a Gooch crucible, and the precipitate washed three or four times by decantation with hot water containing acetic acid and ammonium chromate. The precipitate of barium chromate is finally transferred to the crucible, and washed with warm water until the filtrate gives but a faint coloration with silver nitrate; the barium chromate is gently ignited and weighed. To estimate the strontium in the filtrate, this is evaporated to dryness, a little water added, and the solution rendered neutral or faintly alkaline by means of ammonia. A little ammonium chromate is next added, and the solution taken almost to dryness. When cool, the residue is treated with 50 per cent. alcohol, which is then decanted through a Gooch crucible, and this treatment repeated several times; finally, the strontium chromate is transferred to the crucible, and washed with strong alcohol. The crucible is dried at 80° to 90° C. and weighed.

The above method may be made volumetric as follows: The solution, containing perhaps 0·3 grm. combined barium and strontium, is diluted to about 300 c.c., heated to boiling, and acidified with 10 to 15 drops of acetic acid, and then there is added, drop by drop, 50 c.c. of $\frac{N}{5}$ potassium bichromate, which has been exactly converted into the normal chromate by the addition of ammonia. When cool, the mixture is made up to 500 c.c. and filtered. To 100 c.c. of the filtrate potassium iodide and hydrochloric acid are added, and the liberated iodine is determined by means of thiosulphate; the difference between the amount of iodine thus found and that corresponding to 10 c.c. of $\frac{N}{5}$ bichromate is the measure of the barium. Another 350 c.c. of the filtrate is evaporated nearly to dryness, a further 10 c.c. of $\frac{N}{5}$ bichromate is added, and the solution neutralised and evaporated nearly to dryness; the residue is triturated with 50 per cent. alcohol, and transferred with more 50 per cent. alcohol to a 250 c.c. flask, which is filled to the mark. The contents of the flask are shaken from time to time during half an hour, and then filtered. To 100 c.c. of the filtrate potassium iodide and hydrochloric acid are added, and the liberated iodine is determined by means of thiosulphate, and calculated back to chromic acid. The difference between the amount of chromic acid thus found and that present in solution before the treatment with alcohol rendered the strontium chromate insoluble is the measure of the strontium. Finally, calcium is precipitated as oxalate in the filtrate from strontium chromate, and estimated volumetrically by means of permanganate.

G. C. J.

Racemic Acid as a Reagent (Precipitant of Calcium and Strontium). A. Kling. (*Bull. Soc. Chim.*, 1911 [iv.], **9**, 355-361.)—When a 10 per cent. solution of racemic acid is added to even very dilute (1 : 10,000) solutions of calcium or strontium acetate, a visible crystalline precipitate forms in a few seconds. Though barium racemate is but sparingly soluble, even a 5 per cent. solution gives rise to no visible crystals under several hours when tested in a similar manner. Racemic acid is therefore a delicate test for calcium or strontium in presence of much barium. The reaction cannot be made use of to separate calcium and strontium from barium, as some barium is always carried down with the racemate of calcium and strontium.

The reaction, is however, of service for the estimation of calcium in presence of aluminium, iron, magnesium, phosphoric or citric acid. To the solution, which should preferably contain 0·2 to 0·3 grm. calcium in 100 c.c., 10 c.c. of an acid solution of sodium acetate (10 per cent. sodium acetate, 10 per cent. acetic acid) is added, and this is followed by 10 to 20 c.c. of a 10 per cent. solution of racemic acid. Precipitation is complete within half an hour, at the end of which period the precipitate is filtered off, washed with cold water, and either ignited and weighed as oxide, or dissolved in hot dilute sulphuric acid, and titrated against permanganate. In presence of iron or aluminium the precipitation is incomplete, unless a large excess of alkaline racemate or tartrate is present. The author adds ten to fifteen times as much sodium tartrate as corresponds to the whole of the iron and aluminium before adding the racemic acid. In presence of iron, aluminium, or magnesium, the precipitate is liable to contain notable traces of these elements, and resolution in hydrochloric acid and re-precipitation by sodium acetate and a little racemic acid is necessary to obtain the best results, which are accurate within 1 per cent.

Strontium may be estimated similarly in presence of phosphates and citrates, but not in presence of much iron and aluminium, which render the precipitation far from complete, even when large additions of sodium tartrate are made.

G. C. J.

Estimation of Very Small Quantities of Bromine in Presence of Chlorides and Iodides. H. Baubigny. (*Bull. Soc. Chim.*, 1911 [iv.], **9**, 352-354.)—The author refers to his own earlier work (*Compt. rend.*, 1897, **125**, 527, 607; 1899, **128**, 1160, 1236) in refutation of Clausmann's contention (ANALYST, 1911, 241) that small quantities of bromine cannot be estimated accurately by gravimetric methods. For quantities of the order of 0·01 mgrm., a colorimetric method such as Clausmann's must necessarily be employed; but, if sufficient material is available, the author would prefer to take 10 grms., or even more, if such quantity rendered the use of a gravimetric method possible. For quantities of the order of 5 mgrms., the author's method gives results which are never above the truth, and never more than 0·1 mgrm. low.

G. C. J.

Estimation of Chlorides, Chlorates, and Perchlorates in the Presence of Each Other. Marqueyrol. (*Ann. Chim. anal. appl.*, 1911, **16**, 167-168.)—The method is based upon the fact that hot nitric acid liberates chlorine quantitatively from chlorides, and decomposes chlorates with the formation of nitrates, but does not affect perchlorates under the same conditions. Chlorides are detected in the usual way, whilst the presence of chlorates is indicated by the yellow coloration of the liquid and characteristic odour of the chlorine vapours produced on introducing a little of the substance into strong sulphuric acid. In testing for perchlorates the solid substance is gently heated on a sand-bath with an excess of pure nitric acid (sp. gr. 1·33 to 1·38) to decompose chlorides and chlorates, the dry residue fused with sodium carbonate and potassium nitrate, the mass dissolved in dilute nitric acid, and the solution tested with silver nitrate. For a quantitative estimation the chloride is determined in the usual manner. The total chlorine is then estimated in the mass obtained by fusing the original substance with ten times its weight of a mixture of sodium carbonate and potassium nitrate; and finally the chlorine of the perchlorates is estimated in the residue left after destruction of the chlorides and chlorates with nitric acid. C. A. M.

Estimation of Copper in Pyrites and in Burned Pyrites. W. N. Iwanow. (*Chem. Zeit.*, 1911, **35**, 551.)—A portion of the copper in burned pyrites obstinately resists solution by means of nitric acid, but the whole of the copper in either pyrites or burned pyrites may be obtained in solution, suitable for electrolysis, as follows: The finely ground sample (3 grms.) is mixed with an equal weight of reduced iron, and the mixture placed in a thin-walled glass tube, 7 cm. × 1 cm. and closed at one end, and covered with a little reduced iron. The tube and its contents are maintained at a low red heat for a few minutes by means of a Bunsen burner. The still hot tube is placed in a 250 c.c. Erlenmeyer flask and cracked by means of a few drops of water. When cool, 50 c.c. of hydrochloric acid are added and the flask placed on the water-bath for half an hour, or until the iron is wholly dissolved. Some of the copper will separate as sulphide, and the rest is precipitated by means of hydrogen sulphide. The separated copper sulphide is filtered off with the sand, etc., washed, and dissolved in nitric acid. The solution is neutralised with ammonia and electrolysed, with or without separation of the iron. The results agree well with those obtained by Lunge's method, and are sometimes higher. G. C. J.

Valuation of Higher Oxides of Lead. A. Chwala and H. Colle. (*Zeitsch. anal. Chem.*, 1911, **50**, 209-249.)—The authors have experimented with all the many iodometric methods which have been proposed for the valuation of minium and lead peroxide, and conclude that these methods should be abandoned. The sources of error in several of them have been fully traced, and it is shown that satisfactory results cannot be expected even with red lead, which lends itself better to estimation by these methods than does the peroxide, unless the titration takes place in strongly acid solution. It is also shown that the presence of acetates of the heavy metals may lead to notable errors, and generally that methods which depend on the liberation of iodine from iodide by strong oxidising agents in presence of organic substances are untrustworthy.

The method of Lux is much more trustworthy, but it occasionally gives rise to low results, the cause of which has been traced to too long boiling with nitric acid. Better results are obtained by the following method, which avoids this source of error, and dispenses with the use of sulphuric acid. To 1 to 2 grms. of the sample, 100 c.c. of $\frac{N}{1}$-nitric acid, and 25 c.c. of $\frac{N}{1}$-oxalic acid are added, and the mixture is heated to boiling for ten to fifteen minutes, or until decomposition is complete. The solution is then titrated with $\frac{N}{1}$ or $\frac{N}{5}$ permanganate (at 70 to 80° C.), which is added as quickly as possible, a preliminary experiment to determine the approximate quantity facilitating this. G. C. J.

Estimation of Lead in Alloys containing Antimony and Tin. A. G. Blakeley and E. M. Chance. (*J. Soc. Chem. Ind.*, 1911, **30**, 518-519.)—The authors have adopted a method combining the procedure of Rossing (see Treadwell and Hall, Second Edition, II., p. 220) for the separation of the lead and other insoluble sulphides, with A. H. Low's dichromate method ("Technical Methods of Ore Analysis," Third Edition, p. 331), after converting to sulphate and dissolving the lead salt in sodium acetate solution.

Half a gram of the finely divided alloy (or an amount containing 0·3 grm. lead) is dissolved in the minimum quantity of aqua regia, or 5 : 1 hydrochloric acid and potassium chlorate, and the solution evaporated to a pasty consistency on the water-bath. Five c.c. of strong hydrochloric acid are run in, the mixture warmed, and 15-20 c.c. of water and a few crystals of tartaric acid added. The liquid is treated with a slight excess of sodium hydroxide solution (20 per cent.), and any precipitation disregarded. After now adding 50 c.c., or an excess, of sodium sulphydrate (50 per cent. solution saturated with hydrogen sulphide and filtered) the solution is warmed gently on a water-bath, with occasional stirring, during half an hour. The sulphides are flocculent and subside readily. The precipitate is washed by decantation with hot water containing 10 c.c. of the sodium sulphydrate solution per litre, and transferred to the filter, the washing being continued till 15 c.c. of the filtrate give no coloured sulphides on acidification with acetic acid. The filter and precipitate are boiled in a Jena flask with 25 c.c. of nitric acid (2 : 3) until no black particles remain, when 15 to 20 c.c. strong sulphuric acid are added, and the boiling continued till the carbonaceous matter is dissolved and the liquid is of a caramel colour (in about five minutes). After cooling somewhat, 0·1 to 0·2 grm. of potassium permanganate is cautiously added and the heating continued, the addition of permanganate and the heating being repeated till the solution turns slightly pink or green. The solution is again boiled after cooling and addition of 50 c.c. of water, and sulphurous acid or sodium bisulphite solution added till the colour is discharged. The solution is kept hot for fifteen minutes, cooled to room temperature, 25 c.c. alcohol added and filtered. The sulphate is washed with dilute sulphuric acid (10 per cent.), and finally with one quantity of cold water, the precipitate being kept largely in the flask. The precipitate is washed into the flask with a boiling solution of sodium acetate (90 grms. lead-free sodium acetate and 8 c.c. of glacial acetic acid per litre), removing every trace of sulphate from the filter-paper, and the liquid, after addition of more sodium acetate solution, warmed till the sulphate is completely dissolved and a clear solution results. A persistent turbidity indicates an incomplete sulphide separation. Sodium hydroxide (20 per cent. solution) is now added drop by drop till the precipitate just formed does not disappear. If the amount of lead is small, it is best to add phenolphthaleïn and then the alkali till a pink colour is produced. Acetic acid is then added cautiously till the solution just clears or the pink colour is discharged. Ten c.c. of potassium dichromate (10 per cent.) is next added, the liquid boiled gently for one or two minutes till the deep orange precipitate settles on removing the source of heat. The liquid is filtered and the precipitate washed ten times with 5 to 6 c.c. each of a hot dilute sodium acetate solution (50 c.c. cold saturated solution diluted to 1 litre). The precipitate is now again washed back into the flask with dilute hydrochloric acid (1 : 2), and the washing continued till all traces of lead chloride are dissolved and no colour remains on the filter. The filtrate is diluted with water to about 300 c.c., 2 c.c. of potassium iodide (50 per cent. solution) added, the solution gently mixed, and the free iodine titrated immediately with $\frac{N}{10}$ sodium thiosulphate solution, using starch-water as indicator. The thiosulphate is standardised on pure lead foil, treated with nitric and sulphuric acids as before, and the sulphate dissolved, etc., as described. Bismuth in small amounts does not interfere with the accuracy of the method, as it remains in solution as sulphate when the lead sulphate is filtered off. Larger amounts (several units per cent.) of bismuth are liable to cause the contamination of the lead sulphate with an indefinite quantity of basic sulphate of bismuth. The bismuth may be detected during the neutralisation of the acetate solution by the failure of the white precipitate to re-dissolve in excess of acetic acid (see Low's "Ore Analysis," Third Edition, p. 334).

A. R. T.

Rapid and Accurate Method for Estimation of Manganese. F. J. Metzger and L. E. Marrs. (*J. Ind. Eng. Chem.*, 1911, **3**, 333-335.)—Hillebrand pointed out (*U.S. Geol. Survey. Bull.*, No. 422, 162) that manganese was a disturbing factor in the determination of ferrous iron in rock by the sulphuric-hydrofluoric acid method, and that the (positive) error was highest when much hydrofluoric acid was present. This suggested to the authors that, in presence of sufficient hydrofluoric acid, manganese might react quantitatively with permanganate, and this is found to be the fact, all the manganese after the reaction being proved to be in the trivalent form. The value of the permanganate solution in terms of iron multiplied by 0·7868 gives, therefore, the value in terms of manganese. The titration is made in presence of 10 c.c. sulphuric acid (1 : 2), 25 c.c. of hydrofluoric acid and 5 grms. of ammonium fluoride. The total volume of the solution should be 100 to 150 c.c., and convenient beakers for the titration may be made by cutting off the tops of the ordinary ceresin bottles in which hydrofluoric acid is supplied. When a solution of pure manganese sulphate, standardised gravimetrically, is titrated with approximately $\frac{N}{30}$ permanganate, the results are accurate to 1 drop of permanganate, or 1 in 400 when 30 mgrms. of manganese, corresponding to about 20 c.c. permanganate is present. The end-point is slightly masked by the brown colour of manganic salts, if more than 40 mgrms. manganese is present. Ferric salts are without influence on the results, or on the sharpness of the end-point.

The method was applied to twelve analysed samples of steel and two of iron, supplied by the U.S. Bureau of Standards. One gram of each sample was dissolved in 10 c.c. nitric acid (1 : 1), and to the slightly cooled solution 1 grm. of ammonium persulphate was added. When effervescence ceased, the solution was boiled for a few seconds, and then evaporated to dryness, not baked. The residue was taken up in 20 c.c. of sulphuric acid (1 : 2) and 30 c.c. of water, and the mixture boiled until a clear solution was obtained. The cooled solution was transferred to a ceresin beaker, 25 c.c. hydrofluoric acid and 5 grms. ammonium fluoride added, and the titration with permanganate proceeded with. The results were in every case higher—in some notably higher—than those supplied with the samples by the Bureau of Standards, but the authors think their own numbers are to be preferred, as their method involves no precipitation or filtration whereby manganese may be, and, in their opinion, probably is lost when following other methods. If agreement with other workers is more important than absolute accuracy, however, the permanganate can be standardised on an analysed sample.

The use of both nitric acid and persulphate is necessary to oxidise the combined carbon of iron and steel. The graphitic carbon of cast-iron is almost without influence, but the authors filter it off just before adding the hydrofluoric acid and ammonium fluoride, although in only one case has a lower result been obtained on a filtered, as against an unfiltered, solution. As much as 2 c.c. of concentrated nitric acid has no effect on the titration, so that 1 or 2 c.c. of dilute nitric acid may be used to assist solution if the residue has been slightly baked after evaporation. G. C. J.

Estimation of Small Quantities of Manganese, especially in the Ash of Plants or of Animal Organs. G. Bertrand. (*Bull. Soc. Chim.*, 1911 [iv.], **9**, 361-370.)—The organic matter is incinerated at a low red heat until no unburned carbon can be seen. The cooled ash is then moistened with hydrochloric acid, warmed on the water-bath for a few minutes, a little sulphuric acid is added, and the mixture evaporated to dryness and again ignited at a low red heat. The treatment with hydrochloric acid and low temperature of the second ignition secure ready solubility of the manganese in the ash. The treatment with sulphuric acid not only eliminates chlorine—an essential preliminary to the persulphate method of estimation preferred by the author—but the last traces of carbon are more easily burned away in a sulphated ash, the fusion-point of which is usually tolerably high.

For estimating the manganese, the author gives preference to the colorimetric method first described by Marshall (*Chem. News*, 1901, **83**, 76), in which potassium persulphate, with the use of a trace of silver nitrate, is employed to oxidise the manganous salt to permanganate. It is shown, however, that Marshall's directions are not only insufficient, but actually misleading. The author dissolves the plant ashes in 10 c.c. of nitric acid (sp. gr. 1·08), adds 5 drops of a 10 per cent. solution of silver nitrate, and at least 0·1 grm. of potassium persulphate. This quantity of persulphate will oxidise 0·4 mgrm. of manganese without risk of manganese dioxide separating. It is recommended that not more of the plant ash should be taken than corresponds to 1 mgrm. of manganese, as otherwise the coloration will be so intense that the accuracy of the estimation will be impaired. Ammonium persulphate must not be used, nor must the addition of silver nitrate be less than that stated. If less silver be used, much more persulphate is required to attain complete oxidation within a reasonable time. The mixture is heated gently until the pink colour shows no further increase in intensity. The temperature is then raised to boiling to destroy the excess of persulphate, and maintained at the boiling temperature until oxygen ceases to be evolved. The cooled liquid is made up to exactly 10 c.c., and its colour matched by that of a standard solution of manganese sulphate which has been oxidised in a similar manner.

The paper includes directions for the successful application of the lead peroxide method to the estimation of the minute traces of manganese found in living tissues. By either method it is possible to estimate quantities of manganese from 0·01 mgrm. to 1 mgrm. with an error not exceeding ±5 per cent. G. C. J.

Exact Method for the Electrolytic Estimation of Metals. W. H. Perdue and G. A. Hulett. (*J. Phys. Chem.*, 1911, **15**, 147-154; through *Chem. Zentralbl.*, 1911, I., 1527.)—To obviate the errors caused by particles of the electrolyte being carried down with a precipitated metal, the use of mercury as a cathode is recommended. The metal is then weighed in the form of an amalgam. The formation of a thin layer of liquid between the platinum dish and the mercury is prevented by a preliminary amalgamation of the platinum. In the case of metals which are only sparingly soluble in mercury, a certain proportion of the precipitated metal may be washed out of the mercury again, and may then take up particles of the electrolyte. Rotation of the mercury cathode will prevent this. C. A. M.

Detection of Nitrites in Water. — Dané. (*Bull. Soc. Chem.*, 1911 [iv.], **9**, 354-355.)—It is proposed to make use of the indole reaction for the detection of nitrites. The reagent employed is a solution of 0·02 grm. synthetic indole in 150 c.c. alcohol (95 per cent.). Of this solution, 2 to 5 c.c. is added to a measured volume of the water to be tested, to which dilute sulphuric acid is next added. If the water contains as much as 0·4 part per million of nitrous acid, the well-known pink or red colour will appear within a minute, and it is said that the reaction may be made the basis of a quantitative colorimetric method for the estimation of nitrites in water. G. C. J.

Modification of the Diphenylamine Test for Nitrous and Nitric Acids. W. A. Withers and B. J. Ray. (*J. Amer. Chem. Soc.*, 1911, **33**, 708-711.)—The authors show that considerable variations exist in the manner of applying this test, in the strength of the reagent used, the amount of sulphuric acid, and the temperature of the reaction. All these factors affect the result. The presence of hydrochloric acid, as is well known, increases the delicacy of the reaction. The following mode of operating is stated to be very delicate: The reagent is prepared by dissolving 0·700 grm. diphenylamine in a mixture of 60 c.c. of strong sulphuric acid and 28·8 c.c. water. After cooling, 11·3 c.c. of strong hydrochloric acid (sp. gr. 1·19) are slowly added, equal to 5 per cent. HCl in the reagent. On standing overnight some of the base separates, proving saturation. One c.c. of the liquid to be tested is well mixed with 1 drop of the above reagent; 2 c.c. of strong sulphuric acid is next run in, in such a way as to form two layers of liquid. The tube is gently shaken to cause slight mixing of the liquids at the point of contact, when it is placed in a water-bath at 40° C. for fifteen to twenty minutes. The test shows the presence of 1 part of nitrite nitrogen in 25,000,000, or 1 part of nitrate nitrogen in 35,000,000. By prolonging the heating to one hour, the test will show 1 part in 32,000,000 or 1 part in 44,000,000 of nitrite and nitrate nitrogen respectively. A. R. T.

Effect of Ignition on Solubility of Soil Phosphates. G. S. Fraps. (*J. Ind. Eng. Chem.*, 1911, **3**, 335.)—Stewart (*Illinois Experiment Station, Bull.* 145) has proposed to estimate the organic phosphoric acid in soils by determining the increase in the quantity of phosphoric acid soluble in 12 per cent. hydrochloric acid, brought about by igniting the sample. It is now shown that certain phosphatic minerals (*e.g.*, wavellite and variscite) only yield 20 to 25 per cent. of their phosphorus to acid under the conditions prescribed by Stewart—namely, twenty-four hours' treatment in the cold—whereas after ignition they are wholly, or almost wholly, soluble.

G. C. J.

Decomposition of Silicates by Fusion with Boric Acid as applied to Qualitative Analysis. E. Rupp and F. Lehmann. (*Chem. Zeit.*, 1911, **35**, 565.)—As a preliminary to a merely qualitative analysis, the method of Jannasch (*Zeitsch. anorg. Chem.*, 1896, **12**, 208) may be much simplified, in particular by omitting the esterification and volatilisation of the boric acid. The authors do not prepare boric anhydride as Jannasch does, but use ordinary boric acid. About 5 to 6 parts of this are mixed with 1 part of the finely divided silicate and the mixture heated in a platinum crucible. As soon as most of the water has escaped, the crucible is covered, and the heating, in a blow-pipe flame, continued for twenty-five to thirty minutes. The product of the fusion is then distributed over the walls of the crucible by giving this a rotatory movement with the tongs. As soon as the fused mass ceases to be fluid, the crucible is partially immersed in water. The sudden contraction causes most of the substance to detach itself from the walls of the crucible, and it is next transferred to a beaker and boiled with 30 c.c. of dilute hydrochloric acid. Only a trace of silica remains undissolved. On cooling, most of the boric acid crystallises out and is filtered off with any other insoluble matter and rejected. It may contain lead chloride, but this is unimportant, as lead, if present, will be detected in the filtrate. The filtrate is evaporated to dryness and dried for an hour. The residue is treated with 40 to 50 c.c. of warm water acidified with hydrochloric acid and the solution filtered from the silica and a second crop of boric acid crystals. The filtrate is submitted to analysis in the usual manner, due regard being paid to the following facts: A single evaporation does not separate silica completely, so that any precipitate apparently of bismuth or aluminium should be further identified. In presence of boric acid, metals of the alkaline earth group may find their way into the precipitate given by ammonium sulphide, but the borates of the alkaline earth metals are readily soluble in a hot solution of ammonium chloride, and by means of this reagent may be separated from the sulphides of the ammonium sulphide group.

G. C. J.

Assay of Silver by Means of the Touchstone. A. Steinmann. (*Ann. de Chim. anal. appl.*, 1911, **16**, 165-167.)—Accurate results may be obtained with the touchstone if the streaks produced by the sample of silver alloy under examination be tested with the nitric acid reagent described below, and the results be compared with those given by the streaks produced by different alloys of known composition. This reagent consists of 40 c.c. of nitric acid (sp. gr. 1·27), 50 c.c. of glacial acetic acid, and 50 c.c. of water. The speed with which this liquid attacks the trace of metal upon the stone enables a close estimation of the proportion of silver to be made. The action of the acid should be arrested by the application of a roll of filter-paper as soon as the streak has become yellowish-brown, without being blackened, and the result is then compared with those given by the standard alloys. In this way it is easily possible to distinguish between silver containing the French legal proportion of 800 mgrms. of silver per grm., and that containing only 795 mgrms. In the case of copper-silver alloys of 900 mgrms. of silver per grm., the surface is thus shown to be of the same composition as the interior, whereas in alloys containing 935 mgrms. the surface is poorer in silver. Alloys of silver with zinc or cadmium are attacked more rapidly by the reagent than copper-silver alloys; but here, also, the proportion of silver may be closely estimated by comparing the results with those given by streaks from standard alloys. C. A. M.

Micro-chemical Test for Sodium. W. Lenz and N. Schoorl. (*Zeitsch. anal. Chem.*, 1911, **50**, 263-265.)—The use of a solution of uranyl acetate in acetic acid as micro-chemical reagent for sodium has been suggested before, but it possesses the disadvantage that the reagent, when stored in glass bottles, dissolves sodium from the glass; and when a small drop of such an old solution is allowed to evaporate alone on a microscope slide, the characteristic tetrahedra of sodium uranyl acetate make their appearance. A better reagent is ammonium uranyl acetate: $NH_4C_2H_3O_2.UO_2(C_2H_3O_2)_23H_2O$. When finely divided, this salt dissolves very quickly, and so can be made use of in the solid state. Potassium gives no similar tetrahedra, but salts of potassium must be separated before applying the test, as they tend to prevent the formation of the crystals of sodium uranyl acetate. If sodium and potassium are present in approximately equal amount, the appearance of the crystals is merely delayed, but if the potassium-sodium ratio is equal to or greater than 5 : 1, the sensibility of the reaction is greatly decreased. G. C. J.

Note on the Titration of Ferrous Oxide in Silicates by the Pebal-Dölter Method. M. Dittrich. (*Ber.*, 1911, **44**, 990-991.)—In the Pebal-Dölter method for the volumetric estimation of iron in silicates, after the decomposition of the latter by treatment with hydrofluoric and sulphuric acids, it frequently happens that portions of the powdered silicate remain undecomposed as strongly adherent hard crusts on the bottom of the platinum crucible. This difficulty occurs more frequently with strongly basic minerals poor in silica and rich in magnesia—*e.g.*, with olivine and rocks containing it. The difficulty may be surmounted by increasing the proportion of silica, by adding to the powdered material, before decomposition, a little quartz, not too finely powdered. Quartz is better than precipitated silica, because the latter is dissolved so quickly that its action in preventing the formation of hard crusts is barely appreciable. The quartz, on the other hand, resists the action of the acid for a certain length of time, and thus maintains the powdered sample in a loose condition. J. F. B.

New Test for Ferrous Oxide, and the Separation of Iron from Aluminium. Charitschkoff. (*Chem. Zeit.*, 1911, **35**, 463.)—By means of a solution of naphthenic acid in light petroleum, ferrous oxide may be quantitatively separated from neutral or faintly acid solutions of its salts, so that the aqueous layer gives no reaction with ferricyanides. The upper petroleum layer is coloured a chocolate-brown if ferrous salts be present. The reaction is as sharp as in the case of copper salts; ferric salts do not respond to the test. This reaction may form the basis of a separation of iron from aluminium. A neutral or slightly acid solution containing both metals is treated so as to convert the iron into the ferrous salt. The liquid is then shaken with a solution of naphthenic acid in light petroleum, and the two layers are separated, the aluminium being estimated in the aqueous portion. J. F. B.

Analysis of Ferrotitanium Alloys containing much Silicon. W. Trautmann. (*Zeitsch. angew. Chem.*, 1911, **24**, 877.)—These alloys cannot be decomposed by simple fusion with bisulphate, even when the operation is several times repeated. They may, however, be easily decomposed as follows: After roasting the sample for some time in a platinum crucible, hydrofluoric acid is added, and subsequently expelled by heating. The mass is fused with potassium bisulphate, and the resulting melt is readily soluble in warm, not too dilute, hydrochloric acid. For the estimation of silicon, a different method must be chosen, and the following is recommended: The roasted alloy is fused with sodium carbonate and a little nitre, and the melt detached from the crucible by the use of nitric acid. After removal of the crucible, complete solution of everything save silica is effected by addition of hydrochloric acid. Sulphuric acid is then added, and the solution evaporated until it fumes. Hydrochloric acid and water are added, the mixture warmed and the silica washed several times by decantation with dilute hydrochloric acid before bringing it on to the filter.

Attempts to estimate carbon in alloys of this type by direct combustion in a current of oxygen, with or without the use of lead peroxide, led to low results. The following figures are recorded: By direct combustion, 1·1 per cent. carbon; by the use of lead peroxide, 1·4 per cent.; by decomposition in a current of chlorine followed by combustion, 2·9 per cent. G. C. J.

Rapid Estimation of Nickel in Steel. G. Raulin. (*Moniteur Scient.*, 1911 [v.], I., 1, 84-86; through *Chem. Zentralbl.*, 1911, I., 1443-1444.)—The steel (1 grm.) is dissolved in 20 c.c. of hydrochloric acid (1 : 1), and the solution boiled with 10 c.c. of nitric acid, and then diluted to 200 c.c. Ammonia is added in quantity sufficient to change the colour of the solution from yellow to red, but insufficient to produce any precipitation, and a measured quantity of potassium cyanide solution (4·45 grms. KCN and 0·5 grm. NaHO per litre) is next added. The iron is then precipitated by means of ammonia, a further 20 c.c. of 2N ammonia is added, the whole made up to 500 c.c. and filtered. To 250 c.c. of the filtrate, 10 c.c. of a solution of ammonium sulphate (600 grm. per litre) is added, together with 5 drops of a 20 per cent. solution of potassium iodide, and the mixture titrated with silver nitrate solution (5·788 grms. per litre) until a permanent turbidity appears. For steels containing less than 10 per cent. of nickel, 0·24 should be deducted from the percentage of nickel found; this represents the error due to adsorption of cyanide by the ferric hydroxide. The correction becomes less with increasing nickel content, and for commercial nickel (98 to 99 per cent.) is practically zero. The estimation is unaffected by the presence of silicon, manganese, chromium, tungsten, molybdenum, sulphur, or phosphorus. G. C. J.

Volumetric Estimation of Sulphur in Iron and Steel. F. G. Elliot. (*Paper read before the Iron and Steel Institute*, May, 1911, p. 11.)—Though preliminary annealing with cream of tartar causes some brands of iron to evolve the whole of their sulphur on subsequent treatment with acid, this is not the case with nickel-chromium steels and other self-hardening material. A better method is the following: The sample (5 grms.) is mixed with 0·25 grm. of anhydrous potassium ferrocyanide and wrapped in a 9 c.m. filter if the sample is a graphitic iron (otherwise in two papers) and placed in a small porcelain crucible and annealed at 750° to 850° C. for twenty minutes in a closed muffle. After annealing and slow cooling outside the muffle, the drillings should still be covered with charred paper, otherwise the temperature or time of annealing has been excessive. The caked contents of the crucible are transferred to a glass mortar, loosened with a pestle, and then transferred to the evolution flask without being allowed to touch its neck. The evolution flask is connected with a condensing-tube 6 inches by 1 inch, containing about 2 inches of water, and standing in a beaker filled with cold water. A tube from this dips into a flask containing 60 c.c. of cadmium chloride solution, which again connects with another flask containing more cadmium solution. Fuming hydrochloric acid (50 c.c.) is now added, and the contents of the flask are rapidly heated until vigorous frothing occurs, when the flame is lowered. When the stream of gas passing through the cadmium solution begins to slacken, the flame is raised so that the liquid just boils, and boiling is continued so long as any gas is given off. The apparatus is disconnected and excess of standard iodine (1 c.c. = 0·00025 grm. S.) is added to the contents of the first absorption flask (usually nothing is found in the second). After addition of 10 c.c. hydrochloric acid (1 : 2), the excess of iodine solution is determined by means of an equivalent solution of thiosulphate, as usual.

The temperature of annealing is of the greatest importance and, unless it can be controlled accurately, it is necessary to work a standard of known sulphur content with each batch of samples. The use of hydrochloric acid of sp. gr. 1·19 and rapid evolution of gas are also essential to accuracy (*cf.* Kinder, *Stahl u. Eisen*, 1908, 249). G. G. J.

Estimation of Sublimed Sulphur in a Mixture of Different Kinds of Sulphur. Taurel and Griffet. (*Comptes rend.*, 1911, **152**, 1182-1183.)—Flowers of sulphur (or sublimed sulphur) consist of a nucleus of crystalline sulphur soluble in carbon bisulphide, surrounded by an amorphous pellicle insoluble in that liquid. The thickness of this pellicle, and consequently the weight of insoluble sulphur, varies very greatly (from 12 to 40 per cent.) with the mode of preparation and with the age of the sample, the insoluble modification undergoing a progressive transformation into the more stable soluble form. If, however, the granules of sublimed sulphur be immersed in carbon bisulphide, the solvent will penetrate through the pellicle, and dissolve the soluble nucleus, without affecting the proportions of the original envelope. On this fact has been based a method of estimating the proportion of sublimed sulphur in a mixture: Two graduated centrifugal tubes are charged, one with 35 c.c. of light petroleum (or other liquid in which sulphur is not readily soluble), and the other with 35 c.c. of carbon bisulphide, and 15 grms. of the sample under examination are slowly introduced into each of the tubes, which are then closed with the finger and slowly shaken until the sulphur is completely distributed. They are next "centrifuged" for ten minutes at a moderate speed, after which they are allowed to stand for a short time, and the volume of sulphur in each tube read.

C. A. M.

Estimation of Total Sulphur in Coal. T. S. Warunis. (*Zeitsch. anal. Chem.*, 1911, **50**, 270-272.)—The coal (1 grm.) is mixed with 2 parts copper oxide and 1 part anhydrous sodium carbonate, and the mixture placed in a porcelain crucible. This is supported in an inclined position in an opening in an asbestos plate, large enough to let the crucible pass half through. The crucible and its contents are heated gradually until the under-side of the crucible attains a red heat, using a Bunsen burner or, for exact work, a spirit lamp. The mixture is stirred from time to time for two hours, and then turned out into a beaker containing bromine water. The crucible is washed with dilute hydrochloric acid containing bromine, and the solution added to the contents of the beaker. More hydrochloric acid is added, and the solution boiled to expel bromine, filtered from coal ash, and the sulphate estimated as barium sulphate as usual. The results are occasionally higher than those obtained by Eschka's method, but always agree well with those obtained by Sauer's method (combustion in a current of oxygen in a combustion tube, containing 3 inches of platinised asbestos, and absorption of the gases by hydrogen peroxide or hydrochloric acid and bromine). G. C. J.

Behaviour of Uranium Phosphates towards Indicators. E. Starkenstein. (*Biochem. Zeitsch.*, 1911, **32**, 235-242.)—In the titration of orthophosphates with uranium acetate, either cochineal or potassium ferrocyanide may be used as the indicator; as soon as all the phosphoric acid has been precipitated as uranium phosphate, the addition of a further quantity of uranium acetate yields a brown coloration with potassium ferrocyanide and a green coloration with cochineal. In the case of cochineal, however, the titration solution must not contain too great a quantity of free acetic acid. Cochineal may be used as the indicator in the titration of glyceryl phosphate, but not potassium ferrocyanide, as this combines with the uranium acetate before the latter reacts with the phosphoric acid of the compound. In the case of pyrophosphates, the titration with uranium acetate yields the theoretical amount of phosphoric acid when cochineal is employed as the indicator, but only one-half the amount of phosphoric acid is found when potassium ferrocyanide is used; the same applies to the titration of inositol phosphate. Only one-half of the phosphoric acid in metaphosphates is titrated when cochineal is used as indicator, and with these salts potassium ferrocyanide cannot be used.

W. P. S.

APPARATUS, ETC.

New Method of Chemical Analysis. J. J. Thomson. (*Discourse delivered at the Royal Institution*, April 7, 1911; through *Nature*, 1911, **86**, 466-469.)—The positive rays with which the author has been working for the last few years afford a means of gaining an insight into the nature of the gases present in a vacuum-tube, and render possible a chemical analysis capable of determining whether, for example, oxygen is present, and, in addition, whether it is in the atomic or molecular condition, and if O_3, or other complex aggregations also occur. The rays after passing through a fine tube in the kathode, are exposed simultaneously to magnetic and electric forces, the former so arranged as to produce a vertical, and the latter a horizontal, deflection of the rays. When neither force is acting, the rays strike a point, *O*, on a screen placed at right angles to their direction, but under the influence of both fields strike a new point, *P*. The length of the vertical line *PN* is equal to the deflection produced by the magnetic field, and the horizontal line *ON* to that of the electric field.

It is known that $PN=A\frac{e}{v}$ and $ON=B\frac{e}{mv^2}$ where *A* and *B* are constants, depending on the strength of the fields and the geometrical data of the tube, *e* is the charge on the particle, *m* its mass, and *v* its velocity, then $\frac{m}{e}=\frac{A^2}{B}\frac{ON}{PN^2}$. When these rays strike a photographic plate it is affected, and on development yields a permanent record of their deflection (*cf. Phil. Trans.*, February, 1911). From the values obtained by measurement of the plates for *ON* and *PN* can be determined the value of $\frac{m}{e}$, which for the same type of carrier is a constant, and this value can be compared for different rays simply by measuring the values of *ON* and *PN*. The locus of *P* is a parabola and not a point, because the rays are not all moving with the same velocity, the slower ones suffering greater deflection. Each type of carrier produces its own line on the plate, and there are as many curves on the plate as there are kinds of carriers; from an inspection of the plate can be found, not only the number of kinds of carriers, but, from the dimensions of the curves, there can at once be calculated their atomic weights. It is in this last respect that the great advantage the new method has over spectrum analysis is obvious.

The author gives as an illustration the photographs of the positive-ray spectrum for nitrogen prepared from the atmosphere, and that from some nitrogenous compounds; in the former there is a line absent in the latter whose $\frac{m}{e}$ value is forty times that of hydrogen. Atmospheric nitrogen, therefore, contains an element of atomic weight 40 (argon). The positive-ray method will detect quantities of a gas too minute to produce any indication in the spectroscope, traces of helium, *e.g.*, having been thus observed. Although but a few months old, the method is already capable of determining with an accuracy of 1 per cent. the atomic weight of a gaseous substance, only $\frac{1}{100}$ mgrm. of the material being required. Impurities in the materials simply produce additional curves, and do not affect the parabola due to the substance under examination, and therefore introduce no error in the determination of its atomic weight.

The method would appear to be peculiarly suitable for the determination of atomic weights, not merely of the emanation from radio-active substances, but also those of the products into which they disintegrate. The rays are registered within a millionth of a second of their formation, and may therefore disclose the existence of intermediate forms of matter having only a transient existence, and so lead to a clearer insight into the processes of chemical action. The following are two out of several photographs described fully in the original paper; the first is that obtained from atmospheric nitrogen, the measurements showing that the atomic weights of the carriers producing the curves were as follows:

Positive.	Negative.
1·00 H +	1·00 H −
1·99 H_2 +	11·2 C −
6·80 N + +	15·2 O −
11·40 C +	
13·95 N +	
28·10 N_2 +	
39·00 Arg +	
100·00 Hg + +	
198·00 Hg +	

H + denotes that the carrier is an atom of hydrogen with one charge, H_2 + a molecule of hydrogen with one charge, N + + an atom of nitrogen with two charges, and so on. Nitrogen prepared from NH_4NO_2 had the following lines:

Positive.	Negative.
6·10 C + +	44·2 CO_2 +
7·02 N + +	65·5 Hg + + + ?
12·08 C +	100 Hg + +
14·01 N +	204 Hg +
27·90 N_2 +	

The spectrum of CH_4 contains a small region with five lines very close together; measurements show that their atomic weights are 12, 13, 14, 15, and 16, corresponding to C, CH, CH_2, CH_3, and CH_4. The author believes that this is the first occasion when the atoms (*sic*) CH, CH_2, and CH_3 have been observed in a free state. A gas through which an electric discharge is passing is a very complex thing, even in the case of an elementary substance like oxygen, in which the lines show that there are present simultaneously—

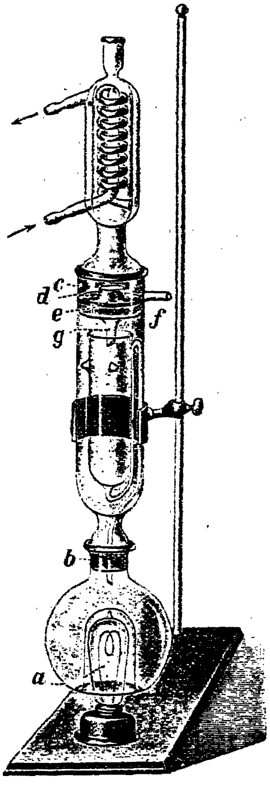
(1) Ordinary molecular oxygen, O_2.
(2) Neutral atoms of oxygen, O.
(3) Atoms of oxygen with 1 positive charge, O +.
(4) ,, ,, 2 positive charges, O + +.
(5) ,, ,, 1 negative charge, O −.
(6) Molecules of oxygen with 1 positive charge, O_2 +.
(7) Ozone with a positive charge, O_3 +.
(8) O_6 with a positive charge, O_6.

And in addition there are free negative corpuscles. An explanation is given of the presence of negatively charged particles, among which have been observed the *atoms* of hydrogen, carbon, oxygen, and chlorine. The two last are not unexpected, but the presence of hydrogen and carbon, usually considered electro-positive elements, is remarkable. No molecules ever occur in the negative side. The paper concludes with a discussion on the corpuscular theory of matter, the view being held broadly that the combination of atoms to form molecules either of compounds or elements fixes corpuscles which were previously mobile, and converts the atoms from conductors of electricity into insulators with a small specific induction capacity.

H. F. E. H.

Control Tests of the Colour Scale used with Schaeffer's Curd Tester. O. von Sobbe. (*Milchwirtsch. Zentralbl.*, 1911, 7, 172-174; through *Chem. Zentralbl.*, 1911, I., 1559.)—The author has made experiments to ascertain whether the quantities of ferric oxide given in Schaeffer's colour-table correspond with the respective shades of colour. Working according to instructions, it was found that after ten minutes the colours did not correspond, but that they gradually developed and were correct after three hours. Since, however, Schaeffer prescribes a time of ten minutes, an error must exist in the use of his table, owing to the incorrect proportions of ferric oxide. According to the author, the table would be correct if five times the prescribed quantities of ferric oxide were taken, or if his numbers, instead of being read as percentages, were taken to represent grms. per 20 grms. of curd.

J. F. B.



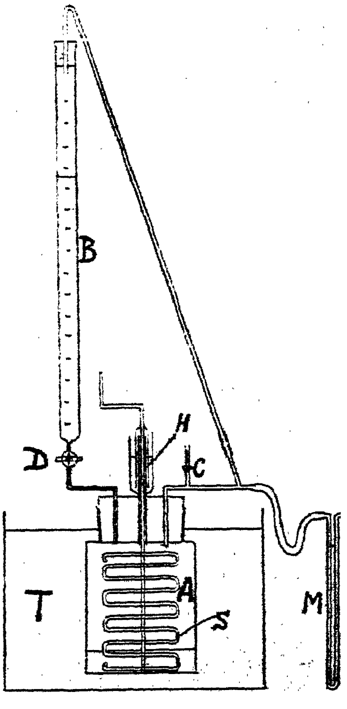
Condenser and Extraction Apparatus. R. Von der Heide. (*Chem. Zeit.*, 1911, 35, 531.)—The condenser and extractor are so constructed that, when the extraction has been carried far enough, the solvent can be distilled off without disconnecting the apparatus. During extraction, the condensed solvent collects in the rimmed plate *d*, and continually overflows through the hole in the raised middle of the plate. When it is wished to distil off the solvent, the condenser is given a quarter turn until the hole *e* (which is in the ground joint of the condenser, not in the plate, as might appear from the sketch) coincides with the outlet *f*, which pierces the wall of the extractor at the joint. The extractor proper with its syphon is removable, and, when it is removed, the remainder of the apparatus may be used as an ordinary reflux condensing apparatus. The new form of flask for electrical heating makes it possible to extract with liquids of relatively high boiling-point, such as water, if the flask be surrounded with an asbestos cylinder.

G. C. J.

Apparatus for Exact Gas Analysis. A. Wilhelmi. (*Zeitsch. angew. Chem.*, 1911, **24**, 870-871.)—Apparatus for exact gas analysis arranged on a single stand, a special feature being arrangements to reduce to a minimum the errors which result from the use of long and comparatively wide capillary connections such as are used with Orsat's apparatus. The apparatus, which is shown in section and fully described in the paper, is made by Eberhardt and Jaeger, Ilmenau. G. C. J.

Measuring Flask for Use in Determining Iodine Values of Oils. M. Dominikiewicz. (*Zeitsch. anal. Chem.*, 1911, **50**, 267-268.)—The neck of the litre flask figured in the paper terminates in a funnel-shaped enlargement. The uppermost part of the cylindrical portion of the neck is ground to accommodate a stopper. After the oil, chloroform, and Hübl solution have been introduced, the stopper is inserted and covered with a 10 per cent. solution of potassium iodide, which renders the flask airtight and absorbs any iodine vapours which may get past the stopper. On lifting the stopper to titrate the contents of the flask, the iodide solution flows in and is washed down by some water from a wash-bottle. The flasks are found useful for other purposes. They are made by Franz Hugershoff, Leipzig.
G. C. J.

Direct Measurement of Changes of Vapour Pressure and Detection of Definite Compounds in Mixed Liquids by its Means. R. Krulla. (*Chem. Zeit.*, 1911, **35**, 471-472.)—The apparatus illustrated serves to follow the changes of vapour pressure when one liquid is added to another in varying proportions. A measured quantity of one liquid (about $\frac{1}{5}$ molar weight) is placed in the closed vessel A, the other in the burette B, the stopcock D of which is closed at first. The temperature of the bath T being maintained at some convenient and constant level, and the cock C being open, the stirrer S is operated by its handle, which works through a mercury seal H. When a state of equilibrium has been reached, the cock C is closed. The manometer M then reads zero. A measured quantity of the liquid in B is run into A through D, and the stirrer again agitated until the level of the mercury in the manometer becomes stationary. Its height is then read. Before proceeding to the next addition of liquid from B, it is convenient to open the cock C and operate the stirrer for a short time, and to commence the next stage of the experiment with the manometer again at zero. G. C. J.



Apparatus for Measuring Radioactivity. B. Szilard. (*Chem. Zeit.*, 1911, 35, 539-540.)—The apparatus described and illustrated in the paper is designed especially with a view to produce an apparatus which, whilst portable, is yet sufficiently sensitive for its purpose. It consists essentially of a magnetic needle pivoted on a vertical point above a scale and in electrical connection with two fixed metallic horseshoe strips about as wide as the needle itself. These horseshoe strips lie in a vertical plane, with their open ends pointing towards the axis of suspension of the needle, and with one arm of each above, the other below, the needle; so that when the box containing the whole apparatus is so placed that this plane is the magnetic meridian, the needle cannot be seen. The needle and strips are insulated from the sides of the box containing them, and this is provided with a glass top and a lens fixed some distance above it, so that the position of the needle at any moment may be observed without error due to parallax. Means are provided for giving a small charge to the needle and strips. In use the apparatus is arranged so that the horseshoe strips just cover the needle; the latter is then charged, which causes it to move out from the meridian; the substance to be examined is introduced into the case on a small sliding shelf provided for the purpose, and the rate of discharge noted. This is compared, as usual, with the rate of discharge when using a known weight of some standard substance, such as uranium oxide. The bottom of the apparatus unscrews, and in its place can be attached a specially constructed condenser suitable for measuring the radioactivity of gases. This also is figured in the paper. The makers are the Laboratoire de Produits Radioactifs, Paris. G. C. J.

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REVIEWS.

A Primer on Explosives for Coal-Miners. C. E. Munroe and C. Hall. Bulletin 423 of the United States Geological Survey. Washington, 1909.

An octavo pamphlet of sixty-one pages, including contents, with twenty-one half-tone plates; it is intended to check the improper use of explosives in coal-mines. Combustion and explosion, and the nature of "low" and high explosives in general and as applied to coal-mining, are simply explained. Squibs, fuse, and detonators, the various modes of coal-mining with explosives, including the proper charges and placing of shots, loading and firing, the advantages of electric firing, and the application of explosives to driving shafts and entries, are dealt with. Throughout, the sources of danger, and precautions for avoiding them, are pointed out. A chapter on the transport and storage of explosives (by B. W. Dunn), a description of the tests for permissible explosives, carried out in experimental galleries containing gas or coal-dust mixed with air, and a list of the publications of the department on the subject, are appended. O. E. Mott.

Allen's Commercial Organic Analysis. Fourth edition. Vol. IV. Edited by W. A. Davis, B.Sc., A.C.G.I., and Samuel S. Sadtler, S.B. 8vo. Pp. viii + 466, with 7 illustrations. London: J. and A. Churchill, 1911. Price 21s. net.

In the volume under consideration a large proportion of the space is given to the essential oils and certain constituents of them, such as the terpenes, camphor and other ketones, but this would appear to be fully justified in view of their commercial and technical importance, as well as the wide scientific interest associated with their more exact chemical investigation. It is not to be expected, however, that even within the space assigned to them, such a large group of natural products, so diverse in character and composition, could be described with the same degree of completeness as in a more specialised treatise on the subject, especially as methods of analysis and tests for the detection of adulterants and impurities must likewise be accorded the consideration which their importance demands.

In presenting a survey of the contents of the present volume, or more particularly of the sections devoted to the essential oils and allied subjects, it would seem desirable and of interest to record some general observations, and also to note some details of statement where inaccuracies appear to exist, without, however, attempting to enter upon a more extended discussion, for which the limitations of space would not permit.

The "Hydrocarbons of Essential Oils" are considered in a chapter of twenty-five pages (pp. 163-187), and these are followed by a chapter entitled "Ketones of Essential Oils," which comprises twenty-eight pages (pp. 189-216). A larger section (pp. 217-301), bearing the simple title "Volatile or Essential Oils" is devoted to a broader treatment of the subject, and includes their extraction, composition, general characters, and analysis, the general information under the latter heading being followed by a more detailed description of the methods employed for the detection or estimation of certain compounds. This comprises, for example, the detection of sulphur compounds, estimation of free acids, phenols, alcohols, esters, aldehydes, and ketones, together with the determination of such factors as the iodine value, methoxyl number, optical activity, and refractive index, and tests for some of the more common adulterants. A knowledge of these methods is naturally of very special importance to the analyst, inasmuch as the commercial valuation of a number of essential oils depends, not only on general tests for their identity and purity, but frequently on the quantitative determination of some particular constituent. A chapter entitled "Special Characters of Essential Oils" comprises within the limits of 128 pages (pp. 303-430) a description of the more important individual oils, and this is accompanied by a tabular representation of their principal characters, constituents, and solubilities, which occupies the last thirty pages of the work.

Notwithstanding the general excellence and the many valuable features of the present volume, there are also some apparent defects as well as obvious errors, and it is thought desirable that these should be noted for consideration in a subsequent revision, in order that a work which has achieved so high a reputation may continue to merit the confidence of those who consult it. In the chapter on the "Hydrocarbons of Essential Oils" there appears to be, for example, no reference to the so-called "olefinic terpenes," such as *myrcene*, which occurs in bay oil, sassafras-leaf oil, and the oil of hops, or the isomeric *ocimene*, from *Ocimum basilicum*. On the other hand, under the subject of "Pinene" (p. 180), this terpene is stated to form "a notable proportion of the oil from bay-leaves." It was shown, however, some years ago, that the true bay oil (not laurel oil) contains no pinene, as had previously been asserted, and its presence would therefore be direct evidence of adulteration (*cf.* p. 315). The name Thoms is wrongly printed on p. 189 as *Thorns*, and on p. 210 as *Thomas*.

In the chapter on "Special Characters of Essential Oils" no consideration appears to have been given to such important articles of commerce as the oils of cinnamon and cassia, the oils of wintergreen (*Gaultheria*) and sweet birch, or the oil of bitter almonds, although the comparatively unimportant oils of hyssop and parsley are described. Under "California Bay Oil" (pp. 315, 316) the only information given regarding its constituents is the statement that "the oil contains a hydrocarbon and appears to contain cineol." It has thus here been overlooked (*cf.* p. 446) that this oil was very thoroughly investigated several years ago, and that its principal constituent, a ketone, $C_{10}H_{14}O$, designated "umbellulone," has been specially studied with respect to its constitution (*J. Chem. Soc.*, 1904, **85**, 629; 1906, **89**, 1104; 1907, **91**, 271; 1908, **93**, 252). Under "Nutmeg Oil" (p. 358) there occurs the statement that "the presence of *myristicol* has been noticed by Gladstone and by Wright," although in the investigation referred to in the paragraph immediately following it was shown that the fraction of nutmeg oil to which the designation "myristicol" had previously been applied consisted of several alcohols, all of which have now been definitely identified. A statement concerning the composition of mace oil (p. 359) is not quite correctly expressed, inasmuch as the crystalline compound obtained by Semmler does not pre-exist in the oil, but was formed by the action of alkalies on the liquid constituent now known as *myristicin*, and, being isomeric with the latter, has been designated *iso*myristicin (*J. Chem. Soc.*, 1907, **91**, 2039).

In the "Table of Essential Oils" (p. 434) there occurs, in connection with the oil of *Asarum Europæum*, the word "asarol," a name which was originally given to the chief constituent of the oil of *Asarum Canadense;* but as this compound has since been found to be identical with *d*-linalool, the latter designation should be adopted (*J. Chem. Soc.*, 1902, **81**, 63). On p. 445 the known constituents of oil of hedeoma (American pennyroyal) are given as hedeomol and pulegone, without consideration of the results of a more recent investigation, which are described on p. 377.

In a section immediately following the above, the name *Hedeoma* is incorrectly applied to the leaves of *Mentha pulegium*, which is known as "European pennyroyal," but the two genera are botanically quite distinct. The statement regarding the constituents of oil of hops (p. 445) should be revised in accordance with the more recent investigation of Chapman (*J. Chem. Soc.*, 1903, **83**, 505), and on p. 450 the statement that the known constituents of oil of nutmeg are "chiefly pinene and myristicin" would suggest some amplification (*cf.* p. 358).

The observations above noted, together with a considerable number of typographical errors, would indicate that in these relatively minor details a little more care might have been exercised. Nevertheless, the intrinsic merits of the work are such that it may be hoped it will receive the recognition accorded to its predecessors, and, in view of the large amount of valuable information it contains, that it will continue to maintain its place as a trustworthy guide in the manifold operations of chemical analysis and investigation.

F. B. Power.

RESINS, INDIA-RUBBER, ESSENTIAL OILS.

Dr. M. B. Blackler is the author responsible for the section on resins, with which this volume opens. Those familiar with this subject know that, with a few exceptions, a particular resin cannot be identified by ordinary analytical processes alone, and consequently the analyst who is faced with a problem of this kind has to base his judgment on a great variety of factors, and must as a rule create his criteria from his knowledge of the properties and characters of resins as a whole. Dr. Blackler has kept this fundamental difficulty in mind, and has written and arranged his matter with a view to enabling his readers to obtain a clear idea of the differences which distinguish the chief groups of resins from each other. In the present nebulous condition of resin analysis this is undoubtedly the best method of procedure.

The errors of the article are for the most part inherited from previous editions of this work. On p. 6 it is implied, without any qualification, that abietic acid contains two carboxyl groups; whilst on p. 23 it is correctly pointed out that this view is erroneous, and that the acid is monobasic. The table on p. 6 includes "alban," one of the resins of guttapercha, as a "resene," although some years ago Van Romburgh and Cohen proved that guttapercha alban is an ester. The misleading information about "anime," including the statement that "it appears in general to be closely related to elemi and copal," is reprinted unchanged from the third edition.

Competent Indian authorities appear to be agreed that all shellac contains orpiment, and not only "some of the dirty yellow shellac of commerce," as the author asserts on p. 67. The third edition stated that French turpentine is called "Gallipot, or Barras," and Dr. Blackler not only repeats this erroneous statement on p. 76, but adds at p. 21 the equally inaccurate assertion that French colophony comes on the the market under the name "galipot." Under "African balsam," p. 83, it is re-asserted that this is probably derived from a Copaifera, whereas it is now perfectly well known that it is obtained from *Daniella thurifera*. The old mistake is made of including the oleo-resin elemi under the "gum resins," and its botanical origin is wrongly given. The statement on p. 17 that a variety of acaroid resin is produced in the Bahamas is incorrect, though the author can no doubt quote good authority for his assertion.

The occasional application of the term "gum" to these products is unfortunate, since it is important, in view of the use of this term in commerce, for such widely different materials as true gums, resins, camphor, opium, and rubber that technical writers should restrict it to its proper application.

Practically all the important resins are dealt with, but descriptions of some of the Euphorbia resins and of gamboge ought to have been included, and as Chinese lacquer is now an article of European commerce, some reference should have been made to this interesting product.

T. A. HENRY.

INDIA-RUBBER.

The article on "India-rubber," which comprises india-rubber, rubber substitutes and guttapercha, is by Mr. E. W. Lewis, A.C.G.I. It covers fifty-five pages, of which roughly forty-two pages are devoted to rubber and five pages to guttapercha. The balance of the article, under the heading Rubber Substitutes, deals with fatty (oil) substitutes, with "recovered" or "reclaimed" rubber, and with certain proprietary articles. In the article on india-rubber, Mr. Lewis deals first with latex and its analysis, and then gives a brief sketch of the work on and our knowledge of the composition and of the constitution of the rubber molecule. This is followed by some notes on the physical and chemical properties of crude rubber. The next section—namely, that on the analysis of crude rubber—is of a practical character, and should be decidedly useful to the general analyst who has no special experience of crude rubber. It might perhaps have been desirable to omit the reference to the Schneider process, or to have made the reference to it of a somewhat briefer character. Mr. Lewis would probably be the first to agree that the method has very little, if any, practical value. The remarks dealing with the vulcanisation of rubber, if somewhat brief, are sound, as one would expect from a chemist of Mr. Lewis's practical experience. Considerable space is devoted to the analysis of vulcanised rubber, and for those who desire further information there are ample references to the literature on the subject. On the whole, this section of the article has been dealt with admirably, but it is necessary for the reviewer to point out that recent work has conclusively shown that pyridine is not a suitable solvent for the separation of tar, pitch, and bituminous matter from many—possibly the majority—of rubber mixings (*cf.* Britland and Potts, ANALYST, 1910, **35**, 539). There are some useful notes on the analysis of ebonite and on the methods for determining the physical properties of rubber. Mr. Lewis deals very briefly with the latter, and also with the mechanical properties of rubber, and, in taking this course, he appears to have been well advised, in smuch as the determination of the physical and mechanical properties of rubber require special apparatus and plant which are not likely to be present in the general analyst's laboratory. Without such special apparatus, attempts to determine the mechanical and physical properties of rubber are likely to do more harm than good, but Mr. Lewis has given references to the literature on this subject for those who desire to pursue it further. On the whole, Mr. Lewis is to be congratulated on handling a difficult and intricate subject in a sound and scholarly fashion.

P. SCHIDROWITZ.

THE MICROSCOPICAL EXAMINATION OF FOODS AND DRUGS. By HENRY GEORGE GREENISH, F.I.C., F.L.S., Professor of Pharmaceutics to the Pharmaceutical Society. London: J. and A. Churchill, 1910. Price 12s. 6d. net.

This is a second edition of Professor Greenish's book, which originally appeared in 1903. A section has been added on fibres, and some additions have been made to the previously existing sections of the work, which is one primarily designed as a working textbook for students. The well-known skill and care of the author as a microscopical expert have resulted in a set of drawings which, as far as they go, afford clear indications of the histological structure of many of the materials with which analysts as well as pharmacists are called upon to deal; but, as only typical articles are selected, the list—and consequently the general utility of the book—is limited. The first section is devoted to starches and the second to hairs and textile fibres, the remaining sections dealing with spores and glands (lycopodium, lupulin, etc.), woods, stems, leaves, flowers, barks, seeds, fruits, rhizomes, and roots, about fifty different typical drugs being dealt with, as well as some of the more common food substances, the analysis of which calls for microscopical examination, some of the more common adulterants of powdered foods and drugs being included. The working instructions are good throughout, being the outcome of long personal experience in this important branch of the analyst's art. BERNARD DYER.

IDENTIFICATION OF THE COMMERCIAL DYESTUFFS. Being Volume III. of a Method for the Identification of Pure Organic Compounds. By S. P. MULLIKEN. New York: J. Wiley and Sons. London: Chapman and Hall. 1910. Price 21s. net.

The introductory chapter refers to the difficulty and complexity of the problem involved in the identification of the several hundred definite chemical species of colouring matters liable to be met with in current industrial dyes. In Chapter II. the author gives directions for the general analytical procedure in the examination of an unknown dyestuff, dividing his tests into four groups: (*a*) Preliminary; (*b*) generic, divisional, and sectional; (*c*) co-ordination; and (*d*) special tests.

The reactions indicated under these headings are likely to prove of service to the analyst who is occasionally called upon to examine commercial brands of dyes, many of which are mixtures of several specific colouring matters.

The tests are arranged in a definite order and are referred to by numbers. The preliminary tests include the identification of indigoid dyes and sulphide colours by vat-dyeing experiments with sodium hydrosulphite and sulphide respectively. The generic tests contain descriptions of the colour-discharge effects produced by Rongalite (sodium formaldehydesulphoxylate) and the regeneration of the colour in certain cases by oxidation with potassium persulphate.

Among the co-ordination tests will be found the effect on dyed fabrics of such general reagents as concentrated sulphuric acid, aqueous sodium hydroxide, nitrous acid, and chromic acid (after-chroming).

The application of the foregoing reactions will generally enable the analyst to refer the colouring matter to its proper class, and special tests are now applied for the purpose of identification. Among these special tests may be mentioned—(i.) The precipitating action of sulphuric acid, sodium hydroxide, tannic acid, and the sulphates of calcium, chromium, and copper; (ii.) the dyeing of wool with different mordants; (iii.) the reduction of azo-dyes with sodium hydrosulphite, and the identification of the fission products; (iv.) the absorption spectra of dye-solutions.

Whenever possible, verification tests should be carried out by making comparative dyeings with the identified dye and standard specimens of the same colouring matter.

The greater portion of the volume is devoted to a tabulation of the commercially important dyestuffs arranged in accordance with their reactions towards the reagents employed in the generic or sectional tests, and indicating the tinctorial effects of the dyes on wool or cotton. The last chapter gives useful suggestions for the identification of dyestuffs in mixtures. G. T. MORGAN.

EIGHTH INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY.

WASHINGTON AND NEW YORK, U.S.A., SEPTEMBER, 1912.

NOTICE.

THE Eighth International Congress of Applied Chemistry will be held in Washington and New York, U.S.A., from September 4 to September 13, 1912, under the patronage of His Excellency the President of the United States. The Hon. President of the Congress is Professor Edward W. Morley, Sc.D., Ph.D., LL.D.; the President, Mr. Wm. H. Nichols, Sc.D., LL.D.; and the Secretary, Dr. Bernhard C. Hesse, 25, Broad Street, New York City.

The Congress is divided into the following sections and subsections:

I. Analytical Chemistry.
II. Inorganic Chemistry.
III*a*. Metallurgy and Mining.
III*b*. Explosives.
III*c*. Silicate Industries.
IV. Organic Chemistry.
IV*a*. Coal Tar Colours and Dyestuffs.
V*a*. Industry and Chemistry of Sugar.
V*b*. India-rubber and other Plastics.
V*c*. Fuels and Asphalt.
V*d*. Fats, Fatty Oils, and Soap.
V*e*. Paints, Drying Oils, and Varnishes.
VI*a*. Starch, Cellulose, and Paper.
VI*b*. Fermentation.
VII. Agricultural Chemistry.
VIII*a*. Hygiene.
VIII*b*. Pharmaceutical Chemistry.
VIII*c*. Bromatology and Pharmacology.
VIII*d*. Physiological Chemistry.
IX. Photo-Chemistry.
X*a*. Electro-Chemistry.
X*b*. Physical Chemistry.
XI*a*. Law and Legislation affecting Chemical Industry.
XI*b*. Political Economy and Conservation of Natural Resources.

Papers will be accepted for reading and discussion in all the above sections, preference being given to those specially adapted for international discussion. They must be sent in not later than July 1, 1912.

A preliminary pamphlet has been issued, and may be obtained from the Hon. Secretary of the British Organising Committee, Charles G. Cresswell, Society of Chemical Industry, Palace Chambers, Westminster, S.W.

June, 1911.

AUGUST, 1911 Vol. XXXVI., No. 425.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

NOTE ON THE COMPOSITION OF BLAUD'S PILL.

By ALBERT E. PARKES, F.I.C., and JOHN D. ROBERTS, B.Sc.

(*Read at the Meeting, May* 3, 1911.)

Blaud's pill, according to the formula recommended by Blaud himself about 1832, was originally prepared from ferrous sulphate and potassium carbonate in equal quantities, compounded with various gums, syrups, etc., to form a workable pill mass, the active constituent being ferrous carbonate, which is formed by the interaction of the two principal ingredients. Since its introduction various alterations in its composition have been made.

At the present time it is incorporated in the British Pharmacopœia as the official *pilula ferri*, and should consist of 150 parts of exsiccated ferrous sulphate and 95 parts of anhydrous sodium carbonate, which are about the theoretical amounts required by the reaction—

$$FeSO_4, H_2O + Na_2CO_3 = FeCO_3 + Na_2SO_4 + H_2O$$

sodium carbonate having been substituted for the original potassium carbonate. The remainder of the pill consists of gum acacia, tragacanth, syrup, and glycerol, the whole being made up into 5-grain pills, each pill containing the equivalent of 1 grain, or about 20 per cent. of ferrous carbonate.

We have recently examined a large number of commercial samples of Blaud's pill, some of which were found to differ considerably in composition from the official formula.

The results of analysis are set out in the table on p. 388, the figures being calculated on the mean weight of five or ten pills.

In numbers 2, 3, 8, 14, 16, 20, 22, 29 and 30, the figures represent the whole pill; in the other cases the pill was examined after removal of its coating. The figures in column 8 represent the amount of SO_3 found in the ash and not in the original pill (except the numbers before mentioned).

Number.	Colour.	Coating.	Ash.	Ash Insoluble in HCl.	Iron as Oxide.	Equivalent to $FeCO_3$ per pill.	SO_3.	Soda, Na_2O.	Potash, K_2O.
			Per Cent.	Per Cent.	Per Cent.	Grain.	Per Cent.	Per Cent.	Per Cent.
1	Pink	Pearl	43·09	18·19	18·18	1·33	0·52	1·38	nil
2	Black	Gelatine	39·71	19·50	15·59	1·33	0·36	0·67	,,
3	,,	Varnish	50·17	19·10	19·15	1·07	13·12	0·90	,,
4	White	Pearl	41·20	14·88	18·52	1·25	1·02	1·05	,,
5	,,	,,	44·36	19·23	19·95	1·38	0·66	1·19	,,
6	Pink	,,	34·42	4·17	17·43	1·01	6·69	—	—
7	White	,,	42·70	17·95	16·47	1·25	0·50	1·68	nil
8	Black	Varnish	46·26	21·70	12·25	1·28	10·96	2·96	,,
9	Pink	Pearl	32·24	3·19	16·76	0·94	5·98	—	—
10	,,	,,	50·57	18·45	18·96	1·32	0·55	0·84	nil
11	,,	,,	33·71	3·89	17·72	0·92	6·29	—	—
12	White	,,	43·08	16·57	20·95	1·50	1·22	1·79	nil
13	Pink	,,	44·95	20·61	17·43	1·30	0·89	0·75	trace
14	,,	,,	52·98	32·70	8·35	0·80	8·78	2·28	,,
15	White	,,	44·54	3·76	12·55	0·89	11·85	—	—
16	,,	,,	57·56	34·80	11·45	1·06	10·32	3·62	nil
17	,,	,,	41·55	2·67	13·10	1·09	13·05	—	—
18	,,	,,	39·55	4·84	19·30	1·46	7·97	—	—
19	Black	,,	40·58	11·10	16·22	1·28	7·04	—	—
20	,,	Gelatine	40·93	1·81	16·69	1·24	16·25	4·34	9·82
21	White	Pearl	38·69	5·49	18·05	1·03	7·15	—	—
22	,,	,,	54·78	19·15	11·85	1·14	10·82	8·91	nil
23	,,	,,	37·94	4·72	18·29	1·13	7·37	—	—
24	,,	,,	41·63	15·45	19·56	1·36	0·99	1·61	nil
25	,,	,,	38·30	4·49	21·08	1·47	7·43	—	—
26	,,	,,	40·24	1·08	15·95	1·00	11·05	1·90	6·95
27	,,	,,	43·25	1·83	16·62	1·13	3·15	—	—
28	,,	,,	38·31	3·34	19·61	1·05	4·21	—	—
29	Black, double strength	Gelatine	47·96	7·59	35·97	2·01	0·54	2·04	nil
30	White	Sugar, Blaud and arsenic	—	0·19	12·74	0·90	11·26	8·80	

It is evident from these figures that many of the samples were made from ferrous carbonate, and in Nos. 3, 8, 13 and 16, the amount of sodium carbonate was very low. The ferrous carbonate was probably first prepared by precipitation with sodium carbonate, and the precipitate, without washing, then made up into pills. This would account for the minute quantities of alkali found in some of the samples. Five samples were found to be deficient in iron, and two samples, it will be observed, contained potassium carbonate.

While it may be held that pills made from ferrous carbonate are quite efficient, it must be admitted that they do not satisfy the requirements of the Pharmacopœia.

NOTE ON THE "PEARL COATING" OF PILLS.

By ALBERT E. PARKES, F.I.C., and JOHN D. ROBERTS, B.Sc.

(*Read at the Meeting, May* 3, 1911.)

In the preceding note it will be observed that the majority of the pills were of the variety known as "pearl coated." In as many cases as possible the coating was removed, estimated, and examined. The approximate composition of the coating and of the pill mass is set out in the following table:

Number of Samples Examined.		Coating.				Pill Mass.		
		Average Weight of Pill.	Amount.	Ash.	Ash insoluble in HCl.	Amount.	Ash.	Ash insoluble in HCl.
		Grm.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
21	Maximum	0·52	40·08	34·75	31·59	78·71	35·70	16·19
	Minimum	0·38	21·29	19·20	17·02	59·92	20·09	0·70

On further examination the ash insoluble in hydrochloric acid was found to consist principally of magnesium silicate, and to have the following composition:

	Coating. Per Cent.	Pill. Per Cent.
Silica and insoluble matter	65·4	71·4
Iron and aluminium oxides	4·4	3·8
Magnesia (MgO)	27·9	21·1
Alkalis	1·7	2·0

The use of magnesium silicate (steatite) for the pearl coating of pills appears to have commenced some twenty-five years ago; of late years it has considerably increased, and at the present time almost every official pill of the Pharmacopœia can be obtained pearl coated.

Opinions differ as the the suitability of steatite as a coating for pills. Gilmour (*Pharm. J.*, 1887, 781) and Cowley (*Chem. and Drug.*, 1895, 923) state that the coating is harmless and easily disintegrated; on the other hand, Tichborne ("Year-Book of Pharmacy," 1901) condemns pearl-coated pills, and refers to them as "bullet-like bodies."

When allowed to disintegrate in water or dilute acid, the coating, under the microscope, has the appearance of transparent, sharp, angular particles, resembling finely-ground glass, and, in our opinion, the ingestion of such inert siliceous matter by persons in delicate health must be attended with grave risks. Quite recently the attention of public authorities has been called to the objectionable habit of polishing rice with mineral matter; the use of the same material as a coating for pills must be regarded with equal disfavour, and, if allowed to be used at all, the amount should be restricted to the smallest possible quantity. The variations in the amounts in the samples examined seem to be far greater than there is any necessity for.

The use of a siliceous material as an excipient in the case of "Blaud's Pill" is quite unnecessary. It will be noticed that in the majority of cases the siliceous matter in the pill mass is small, and can be neglected; it is obviously a difficult matter to free the pill entirely from its coating before examination, but in the other cases it has evidently been added to the pill mass before coating, and attention may also be called to the fact that it is these particular pills which are also deficient in alkaline carbonate.

The British Pharmacopœia sanctions the use of kaolin as an excipient in certain pills, which, however, do not include the one in question.

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THE COMPOSITION OF MILK.

By H. DROOP RICHMOND, F.I.C.

(Read at the Meeting, May 31, 1911.)

In the present communication the results of the analyses of milk during 1910 made in the laboratory of the Aylesbury Dairy Company are given. The average results of 19,282 samples of milk received from the farms is given in the table below:

Average Composition of Milk during 1910.

Month.	Morning Milk.				Evening Milk.				Mean.			
	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.
		%	%	%		%	%	%		%	%	%
January ...	1·0321	12·50	3·62	8·88	1·0318	12·80	3·92	8·88	1·0319	12·65	3·77	8·88
February ...	1·0321	12·39	3·53	8·86	1·0318	12·71	3·85	8·86	1·0319	12·55	3·69	8·86
March ...	1·0320	12·32	3·48	8·84	1·0317	12·64	3·82	8·92	1·0319	12·48	3·65	8·83
April ...	1·0319	12·21	3·41	8·80	1·0316	12·59	3·80	8·79	1·0317	12·40	3·61	8·79
May	1·0322	12·11	3·28	8·83	1·0318	12·58	3·74	8·84	1·0320	12·35	3·51	8·84
June	1·0323	12·11	3·24	8·87	1·0317	12·58	3·76	8·82	1·0320	12·35	3·50	8·85
July	1·0321	12·20	3·37	8·83	1·0315	12·63	3·85	8·78	1·0318	12·42	3·61	8·81
August ...	1·0322	12·37	3·48	8·89	1·0317	12·86	4·00	8·86	1·0319	12·61	3·74	8·87
September ...	1·0324	12·56	3·60	8·96	1·0320	12·98	4·03	8·95	1·0322	12·78	3·82	8·96
October ...	1·0325	12·75	3·75	9·01	1·0322	13·16	4·14	9·02	1·0324	12·96	3·95	9·01
November ...	1·0326	12·89	3·84	9·05	1·0324	13·15	4·11	9·04	1·0325	13·02	3·97	9·05
December ...	1·0323	12·78	3·80	8·98	1·0322	13·04	4·04	9·00	1·0323	12·91	3·92	8·99
Average ...	1·0322	12·43	3·53	8·90	1·0319	12·81	3·92	8·89	1·0320	12·62	3·73	8·89

The usual variations were noticed—that is, the lowest fat occurred in May and June, and the highest in October and November; the drop in solids-not-fat so often found in July and August was scarcely noticed in 1910.

In continuation of the tables published in former years, I give the percentage of the samples falling between each 0·1 per cent. of fat below 3·0; I have extended it this year to include April and July, as quite an appreciable number of samples below the limit were found. In the other months of the year such low samples are practically, if not entirely, non-existent. The table refers to morning milk only, hardly any samples of evening's milk being below 3·0 per cent. of fat.

PERCENTAGE OF SAMPLES OF MORNING MILK FALLING BELOW 3·0 PER CENT. FAT.

	2·9 to 3·0 per Cent. Fat.	2·8 to 2·9 per Cent. Fat.	2·7 to 2·8 per Cent. Fat.	Below 2·7 per Cent. Fat.
April, 1910	1·6	0·3	0·5	0·9
May, 1910	3·0	2·1	0·8	0·5
June, 1910	3·0	1·2	1·0	0·1
July, 1910	0·7	0·7	0·1	—

These results do not greatly differ from those of previous years, but the period when cows frequently yield milk below standard appears to be extending into both April and July.

With reference to the statement that I made last year as to the occurrence of low solids-not-fat during March and April of 1910, which were due to a low percentage of proteins, I give here the composition of the milk from the two farms on which these low solids-not-fat were observed; only the evening milk in each case contained low percentages of solids-not-fat:

	FARM B.						FARM C.		
	March.			April.			March.		
	Average.	Minimum.	Maximum.	Average.	Minimum.	Maximum.	Average.	Minimum.	Maximum.
Total solids ...	12·70	—	—	12·66	—	—	12·54	—	—
Fat	4·24	—	—	4·15	—	—	4·07	—	—
Milk-sugar ...	4·55	—	—	4·52	—	—	4·61	—	—
Proteins ...	3·18	2·96	3·47	3·26	3·11	3·52	3·13	2·99	3·33
Ash	0·73	—	—	0·73	—	—	0·73	—	—
Solids-not-fat ...	8·46	8·20	8·70	8·51	8·30	8·70	8·47	8·15	8·75

In each case about one-half of the samples fell below 8·5 per cent. of solids-not-fat, the bulk of them lying between 8·4 and 8·5; there were only four samples which did not substantially conform to the standard of 8·5 per cent., in which the proteins fell appreciably below the figure corresponding to 0·5 per cent. of nitrogen (3·19 per cent.). These were—

Solids-not-fat ...	8·35	8·30	8·30	8·15
Proteins	2·96	2·96	2·99	3·02

An unusual instance of low solids-not-fat accompanied by a high percentage of proteins was published in the ANALYST (1911, p. 11).

In the last three weeks of December the milk from a farm was found to be constantly below 8·5 per cent. of solids-not-fat. The composition for the month was—

	Average.	Minimum.	Maximum.
Total solids	12·96	—	—
Fat	4·59	—	—
Milk-sugar	3·86	—	—
Proteins	3·72	3·60	3·98
Ash	0·79	—	—
Solids-not-fat...	8·37	8·05	8·95

The acidity of this milk was very low, and averaged 13·4 degrees. Inquiries at the farm showed that the cows were healthy, but were nearly dry, and were only being milked once a day; I learn that this is not an uncommon practice during the winter on farms where cheese is made.

My experience this year has shown that, while the rule that genuine samples containing less than 8·5 per cent. of solids-not-fat should contain 0·5 per cent. of nitrogen and 0·7 per cent. of ash is not an absolute one, the exceptions to this are very few.

DISCUSSION.

Mr. C. REVIS agreed that in the great majority of cases in which deficiency in non-fatty solids occurred it was due to deficiency in milk-sugar. In determining whether a low percentage of non-fatty solids was due to tampering or to abnormality, a determination of the proportion of milk-sugar afforded, in his experience, very useful information, and was well worth the small additional time that it occupied.

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FURTHER ANALYSES OF GHEE.

BY E. RICHARDS BOLTON AND CECIL REVIS.

(Read at the Meeting, May 31, 1911.)

SINCE the publishing of our paper entitled "Some Analyses of Ghee" (ANALYST, 1910, **35**, 343), a number of other samples have come into our hands, and, as some of these are known to be samples of ghee as sold in the market in the City of Bombay, we thought it desirable to place the analyses on record. In publishing these results, we feel it necessary to draw attention to the Reichert-Meissl value of 18·24 given for one sample of ghee made from buffalo milk, and published in a paper by Kesava-Menon (*J. Soc. Chem. Ind.*, 1910, **29**, 1428). We feel certain that this is quite an abnormal figure for true ghee, and, if accepted in any way as a representative figure, would lead to the passing of a considerable amount of adulterated ghee as pure. Of all the samples we have examined, none have lead us to suppose that pure ghee has anything but a high Reichert-Meissl value, usually approaching or even exceeding 30.

We again draw attention to the fact that the process of Avè Lallemant, as referred to in our previous paper, is of distinct value in the examination of ghees:

Source.	Malabar Coast.	Bombay City.					
		A.	B.	C.	D.	E.	F.
Reichert-Meissl value ...	24·4	19·00	25·70	26·20	28·40	29·80	25·60
Polenske value	1·8	0·90	0·90	1·50	1·60	1·40	1·20
Saponification value ...	228·2	219·90	213·80	223·60	224·90	225·40	221·40
Iodine value (Wijs) ...	—	32·37	37·28	29·24	28·11	29·55	31·83
Refractometer index at 40° C. (Zeiss scale) ...	42·7	43·10	44·30	44·80	42·00	42·00	42·60
Valenta number (° C.)...	—	48·50	43·50	42·50	36·00	33·50	50·00
Sp. gr. $\frac{99^\circ}{15^\circ}$ C.	—	0·8635	0·8627	0·8619	0·8656	0·8656	0·8618
Baryta values (Avè Lallemant): (*a*) Total ...	312·0	300·50	292·30	305·60	307·50	308·10	302·70
(*b*) Insoluble	256·7	252·20	256·80	256·20	252·40	251·50	256·40
(*c*) Soluble ...	55·3	48·30	35·50	49·40	55·10	56·60	46·30
$b-(200+c)$ ...	+1·4	+3·90	+21·30	+6·80	−2·70	−5·10	+10·10
Free fatty acids (as oleic)	—	0·37%	2·23%	2·39%	1·83%	1·84%	0·92%
Melting-point (° C.) ...	—	42·50	45·00	43·00	43·00	41·00	43·70
Inferences	Doubtful purity	Adulterated	Highly adulterated	Adulterated	Pure	Pure	Adulterated

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THE DETERMINATION OF THE AMOUNT OF DISSOLVED OXYGEN ABSORBED BY SEWAGE EFFLUENTS CONTAINING NITRITES, AND OF THE AMOUNT OF NITRITE IN SEWAGE EFFLUENTS AND WATER.

By R. W. CLARKE, F.I.C.

(*Read at the Meeting, May* 31, 1911.)

CONSIDERABLE attention has lately been paid to the determination of the amount of dissolved or atmospheric oxygen absorbed by sewage effluents. This has been due in great measure to the fact that, in the fifth report of the Royal Commission on Sewage Disposal, great weight is attached to the amount as a criterion of the purity and non-putrescibility of an effluent.

Two methods of determination are in general use—viz.:

1. Absorption of atmospheric oxygen by means of Adeney's apparatus, the decrease in volume of a known amount of air being measured.

2. Absorption of dissolved oxygen from tap-water, the amount of dissolved oxygen present at the commencement and after definite periods of time being estimated by Winkler's method or one of several modifications thereof. These are referred to in Rideal and Stewart's paper (ANALYST, 1901, **26**, 141).

The Adeney apparatus was found on several occasions to give unsatisfactory results, so it was discarded in favour of purely chemical methods.

In the absence of nitrites, Winkler's method is perfectly satisfactory, and gives concordant results under identical conditions as to dilution and temperature. In the presence of nitrites, however, a definite end-point is not obtainable on titration with thiosulphate, owing to oxidation of N_2O_2 and consequent liberation of a further quantity of iodine, when it is again reduced to N_2O_2, the cycle being repeated, according to the equations:

1. $2HNO_2+2HI=N_2O_2+2H_2O+I_2$.
2. $2N_2O_2+2H_2O+O_2=4HNO_2$.

The method suggested by Rideal and Stewart (*loc. cit.*), while quite satisfactory for the purpose to which it is applied by them, is inapplicable to the effluent before dilution, when the amount of dissolved oxygen absorbed is to be determined, as the whole character of the effluent is, naturally, changed by the oxidising action of the permanganate, and, if applied to the diluted effluent, cannot be relied on completely to oxidise the nitrite, an indefinite end-point being still obtained.

It was found that the difficulty was entirely avoided if free acid was not present. Neutralisation can be effected satisfactorily by adding a small excess of light carbonate of magnesia to the acid liquid. The method may be summarised as follows: The effluent is diluted to a sufficient extent (ten times or more in the case of bad effluents) with good tap-water and thoroughly shaken, and a number of bottles (usually five) of known capacity, approximating to 300 c.c., are completely filled therewith. The amount of dissolved oxygen present at the start, and after one, two, and five days respectively, is determined by adding 1 c.c. of 33 per cent. $MnCl_2$ to the bottom of the bottle by means of a pipette with a long and narrow delivery tube, followed by 3 c.c. of 33 per cent. NaOH containing 10 per cent. KI. The bottle is then shaken, and allowed to stand for some time, atmospheric oxygen being of course excluded. When the mixed hydrated oxides of manganese have settled to the bottom, 3 c.c. of hydrochloric acid are added and the bottle again shaken. A sufficient quantity of $MgCO_3$ to neutralise the free acid is then added, and, after allowing time for the completion of the reaction, the contents of the bottle are poured into a flask and titrated with thiosulphate of known strength (a convenient strength is about $\frac{N}{50}$).

The difference in amount of dissolved oxygen present at the commencement and after a given interval, multiplied by the dilution, gives the amount of oxygen absorbed. The assumption is here made that there is no appreciable change in the amount of nitrites present during the interval. Up to the present all experiments have shown this assumption to be justified.

The possibility of applying the reaction to the estimation of nitrites was at once apparent. Several different methods were tried. The best seems to be as follows: A tall bottle, holding not less than 500 c.c., and having a tightly-fitting stopper, is required. One of the ordinary quart bottles, with spring stopper, such as is used by dairy companies for sterilised milk, is convenient. This is filled with the nitrite-containing water or effluent to within about 20 c.c. of the whole content, the quantity being measured. To this are added 4 c.c. of 33 per cent. $MnCl_2$ and 12 c.c. NaOH and KI solution, as for the estimation of dissolved oxygen. A further quantity of about 1 grm. of KI is added, and the bottle closed and very thoroughly shaken. It is important that a small bubble of air be left in, as the stopper fits sufficiently tightly to cause the bottle to burst on slight increase in temperature if this be not done. The bottle is then allowed to stand at rest overnight. The hydrated oxides of manganese sink to the bottom, leaving a solution of manganese nitrite in water which is free from dissolved oxygen.

A stoppered bottle of known capacity, of about 300 c.c., is then filled with the clear solution by means of a siphon (aeration being avoided), and acidulated with 3 c.c. of HCl; this is then neutralised with $MgCO_3$, and the iodine liberated is titrated with thiosulphate of known strength. A weak solution is best; that used for the determination of oxygen absorbed from permanganate answers excellently. From this result the quantity present in the original water is easily calculated.

Experiments conducted with tap-water containing known amounts of potassium nitrite, varying from 0·04 to 0·6 part per 100,000 of nitrogen, gave results within 5 per cent. of the quantities taken; and the presence of organic matter, such as is commonly present in sewage effluent, appears to cause no error. The long sedimentation of the oxides of manganese is necessary, as the finer particles of manganous hydrate settle down much more slowly than do the more highly oxidised hydrates; and the affinity of manganous hydrate for oxygen is so great that it is impossible to siphon off the liquid without a portion being oxidised, and this causes a liberation of iodine in excess of that due to the nitrite. The other well-known methods of estimating nitrites in the small quantities in which they occur in water and sewage, such as Thresh's, the *m*-diaminobenzene, and the *α*-naphthylamine and sulphanilic acid methods, failed to give such satisfactory results as that above-described. Moreover, the method is extremely simple, and, so far as actual manipulation is concerned, takes very little time, as is also the case with the determination of dissolved oxygen absorbed.

DISCUSSION.

Mr. W. T. BURGESS said that, in connection with the process for the estimation of dissolved oxygen which had been communicated to the Society by Dr. Rideal and himself in 1909, they had observed that, during the time that the effluent was under examination (from one day to five days), the proportion of nitrite present did not sensibly change, and this was confirmed by Mr. Clarke's work. With regard to the dilution of the effluent with ten times its bulk of water, he would point out that the large multiplication of the results which this involved was a decided disadvantage. He thought that as a rule it would be sufficient to dilute with an equal bulk of fully aerated water, provided that the effluent was fully aerated to start with.

Mr. E. HINKS said that he had had an opportunity recently of trying Mr. Clarke's process, and had found that when the solution was neutralised with magnesium carbonate the extension of the end-point by the nitrite was entirely obviated. In the application of the process to the estimation of nitrites he had not been quite so successful. With quantities under 0·1 part per 100,000, his results were always too high, but of course his experience with the process was very limited. One drawback to this part of the process seemed to be that it required so large a quantity of the sample—more than would be likely to be available in ordinary cases after the other determinations had been made.

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THE ESTIMATION OF QUININE AS THE ACID CITRATE IN CERTAIN ORGANIC LIQUIDS.*

By T. COCKBURN, F.I.C., and J. W. BLACK.

(*Read at the Meeting, May* 31, 1911.)

The results of the estimation of quinine in organic liquids, such as urine, by direct extraction of the alkaline liquid with ether, are very unreliable. The quinine thus obtained is contaminated with resinous and colouring matter, and therefore results obtained by direct weighing of the ether extract are entirely erroneous.

Nishi's method (Analyst, 1909, **34**, 443) obviates this source of error, by precipitating the quinine, dissolved in ether, as the acid citrate, by the addition of an ethereal solution of citric acid. That the compound formed is of constant composition and corresponds to the acid citrate is proved by Nishi's published results, the accuracy of which we confirmed by a series of estimations, in which we employed pure anhydrous quinine, with the following results:

Quinine taken.	Acid Citrate of Quinine found by Precipitation.	Percentage of Quinine in Citrate Precipitate.	Quinine calculated from $C_{20}H_{24}N_2O_2.C_6H_8O_7$.	Difference.
Grm.	Grm.		Grm.	Grm.
0·0508	0·0812	62·56	0·0509	+0·0001
0·1069	0·1688	63·32	0·1059	−0·0010
0·1366	0·2144	63·75	0·1346	−0·0020
0·1554	0·2476	62·76	0·1555	+0·0001
0·1758	0·2809	62·58	0·1763	+0·0005
0·2100	0·3394	61·87	0·2131	+0·0031
0·2808	0·4398	63·84	0·2761	−0·0047

The theoretical quinine content of the acid citrate $C_{20}H_{24}N_2O_2.C_6H_8O_7$ is 62·79 per cent. Our average figure is 62·95 per cent., and, considering the small quantities of quinine used, the greatest deviations from this average figure are well within the limits of experimental error. The corresponding figure obtained by Nishi was 62·57 per cent. The general characteristics of the acid citrate were given by Nishi, and our results are in perfect agreement with his. The compound is almost completely insoluble in ether, slightly more soluble in cold water and in alcohol, whilst in hot water it readily dissolves. The process as detailed by Nishi proved cumbersome, as part of the precipitate adheres firmly to the sides of the flask in which the quinine is precipitated, necessitating the weighing of this flask, as well as the Gooch crucible containing the major portion of the precipitate.

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* This work was carried out in accordance with the terms of the "Analytical Investigation Scheme."

We modified the method of collecting the precipitate, whereby the process is simplified, and yields excellent results. The details of the method as simplified by us are as follows: 250 c.c. of the urine are made strongly alkaline with strong sodium hydroxide, warmed and extracted in a separator with ether, the extraction being repeated three times. We found it necessary, in order to lessen the tendency to emulsify, that each extraction should proceed at as high a temperature as is consistent with safety, and that the urine should be strongly alkaline. The ether extracts are collected, washed with small quantities of water, placed in a flask, and the ether evaporated. The residue, consisting of quinine admixed with various impurities, is dried in the water-bath. It is then dissolved in 20 c.c. of anhydrous ether, filtered into a weighed 4-ounce Erlenmeyer flask, the filter-paper and flask being washed with small quantities of ether. With the flask is weighed a filtering-tube, consisting of a small thistle funnel, the bulb of which (diameter 10 mm.) is packed with dry asbestos, the whole being dried in the water-bath. To the filtered ethereal solution is then added 10 c.c. of a saturated ethereal solution of citric acid, and allowed to stand for twenty-four hours. The supernatant liquid is then drawn off through the filter-tube, by means of the usual aspirating apparatus. The precipitated quinine acid citrate, which is usually quite white and free from impurities, is washed three times with 10 c.c. of ether (sp. gr. 0·720) the supernatant ether being drawn off as before. The filter-tube is now placed in the Erlenmeyer flask, the whole dried first at a gentle heat to prevent spirting, and then at 100° C. till constant, and weighed as $C_{20}H_{24}N_2O_2.C_6H_8O_7$.

Known quantities of pure dry quinine were added to urine, and the following results were obtained, using the above method:

Pure Quinine taken.	Quinine Acid Citrate found.	Quinine found.	Difference.
Grm.	Grm.	Grm.	Grm.
0·0306	0·0467	0·0293	−0·0013
0·0499	0·0850	0·0533	+0·0034
0·0682	0·1106	0·0694	+0·0012
0·1000	0·1594	0·1000	±0·0000
0·1093	0·1766	0·1108	+0·0015
0·1406	0·2226	0·1397	−0·0009

The saturated ethereal solution of citric acid is prepared by dissolving citric acid, previously dried *in vacuo*, in anhydrous ether. Care should be taken that the precipitate be not allowed to remain unduly long in contact with the precipitating solution, as thereby high results are obtained; whilst if the ether is not anhydrous, or if the washing of the precipitate with ether is prolonged, the results are too low.

The melting-point of the acid citrate recovered from urine we found to be

194° C., whilst the average of several nitrogen determinations (Kjeldahl) was 5·49 per cent., theory requiring 5·42 per cent.

We applied the method to the estimation of quinine in citrate of iron and quinine, prepared in accordance with British Pharmacopœia requirements, with the following results:

Quinine by Extraction.	Quinine calculated from Acid Citrate.	Difference.
Per Cent.	Per Cent.	Per Cent.
14·47	14·45	- 0·02
14·15	14·07	- 0·08

The process may be employed for the estimation of quinine in the presence of caffeine, but does not afford a means of separation of the common cinchona alkaloids, since all these give a precipitate with an ethereal solution of citric acid.

CORPORATION CHEMICAL DEPARTMENT,
GLASGOW.

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THE ASSAY OF WOLFRAM CONCENTRATE.

By H. W. HUTCHIN.

WOLFRAM is bought and sold on the unit system, taking tungstic oxide as the basis. The average price per unit during the last five years has been 34s. 6d.; the buyers require a minimum of 60 units, although poorer material may be sold, but at a lower price per unit. The necessity for an accurate method of assay of such a valuable product is obvious. As no reliable volumetric method for the estimation of tungstic acid is known, the determination is in all methods made by weighing pure tungstic oxide. Tungstic oxide results from the ignition of either ammonium tungstate, basic mercurous tungstate, or certain organic salts. All three reactions are used in assays. The use of organic salts is so far restricted to the assay of tungsten in alloys, and does not come within the scope of the paper.

Finely powdered wolfram may be decomposed by boiling with strong hydrochloric acid or by boiling with caustic soda solution. With the acid treatment the tungstic acid is liberated and remains undissolved, but with the alkali it passes into solution as tungstate of soda. A preliminary ignition with lime, or calcium carbonate and salt, or calcium carbonate and ammonium chloride, renders wolfram more easily attacked by acids, but may under certain conditions be attended with undesirable complications. Wolfram fused with caustic alkalis or the alkaline carbonates is converted into tungstate of soda or potash, both readily soluble in water.

The use of acid allows of the final determination being made either by ignition of ammonium tungstate or basic mercurous tungstate, but use of alkali necessitates the adoption of the mercury method.

Such reactions are made use of in one or other of the four methods described hereafter, viz.:

1. " Aqua regia–ammonium tungstate " method.
2. " Aqua regia–mercurous nitrate " method.
3. " Soda digestion–mercurous nitrate " method.
4. " Soda fusion–mercurous nitrate " method.

1. "AQUA REGIA-AMMONIUM TUNGSTATE" METHOD, MORE COMMONLY KNOWN AS THE "AQUA REGIA" METHOD.

To 1 grm. of the finely powdered wolfram in an 8-ounce beaker add about 10 c.c. strong hydrochloric acid, and agitate carefully to prevent caking. Add more acid (about 120 c.c. in all), cover with a clock-glass, and boil briskly till the volume of acid is reduced to about 5 c.c. Allow to cool, add 5 c.c. strong nitric acid, and digest at nearly a boiling temperature for from five to ten minutes. Dilute with water to about 100 c.c., allow to settle, and filter through a 9-cm. filter, wash with water, working so as to obtain as little tungstic acid as possible on the filter. To the beaker add about 10 c.c. distilled water and 10 c.c. dilute ammonia (1 : 1), adding the latter in small quantities down the sides of the beaker. With a rubber-tipped rod remove any deposit from the sides of the beaker into the alkaline liquor, raise to the boil, and stir well (if the suspended matter does not readily subside, boil again until the desired result is attained). Allow to settle, and whilst still hot filter through the original paper into an 8-ounce flask. Wash the beaker two or three times with small quantities of distilled water, decanting each time from any heavy mineral should it be present. Complete the washing of the filter-paper, and reserve the cover-glass, filter-paper, and beaker. Evaporate the solution of ammonium tungstate in the flask to a small volume, transfer carefully to a weighed platinum dish, and continue the evaporation to dryness on a water-bath. Ignite the dish and contents gently at first, and more strongly afterwards. Weigh as tungstic acid.

To the beaker containing the heavy residues add 5 c.c. hydrochloric acid, cover with the original clock-glass, and boil till nearly dry; add a few c.c. of nitric acid, and heat again for ten minutes. Dilute with water, allow to stand till the deposit has subsided, filter though a small paper, and wash beaker and paper several times.

Wash the filter-paper, also the original filter-paper, with a little hot dilute caustic soda solution followed by small washes of distilled water, and collect the alkaline liquors and washings in a beaker. If the clock-glass is stained with tungstic acid, dissolve with a few drops of the soda solution and add to the contents of the beaker. Raise the contents of the beaker to boiling, add about 2 grms. ammonium nitrate, and stir well whilst boiling. Filter through one of the papers previously used, and wash a little. Nearly neutralise the filtrate with dilute nitric acid, add a few c.c. of mercurous nitrate solution, stir well, filter on a small ashless paper, wash, ignite, and weigh as tungstic acid. Add this weight to the weight of the tungstic acid in the platinum dish.

Remarks on the Method.—Fine grinding is essential to success. One grm. is a convenient quantity, but with experience 2·5 grms. may be taken without requiring any modification of the quantities given in the assay.

The complete decomposition with the boiling hydrochloric acid takes about one hour, but it is better to continue boiling if the volume of residual liquors is much more than 5 c.c. The addition of nitric acid, with further digestion, is a concession to custom, but it would seem from experiment that in some instances it is unnecessary; for instance, two samples of wolfram gave 71·57 and 75·6 per cent. respectively, using nitric acid, and 71·6 and 75·54 per cent. respectively, using no nitric acid. Departure from custom is not recommended without a more extended series of experiments. In filtering the acid liquors from the tungstic acid, etc., a special wash-water is unnecessary; with water only the filtrate and washings are free from dissolved tungstic acid. The instructions given for effecting the solution of the tungstic acid in ammonia are based on experience; turbid filtrates seldom occur, and the volume of solution for concentration is reduced to a minimum, thereby saving time without sacrificing accuracy. With a turbid filtrate it is better to continue the assay and determine the amount of impurity in the tungstic acid after weighing, as follows: Add sufficient distilled water to cover the tungstic acid in the platinum dish, and about 1 grm. of pure caustic soda. Digest on the water-bath for a few minutes, till all the tungstic acid has dissolved. Wash the contents of the dish into a suitable beaker, add 3 grms. of ammonium nitrate, boil, filter on a small ashless paper, wash, burn, and weigh. Deduct the weight from the original weight of tungstic acid.

The retreatment of the residues from the ammonium tungstate filtrate is the crucial point of the assay; without it concordant results are rare, the residues being seldom free from tungstic acid. Its presence may be due either to unattacked wolfram or to tungstic acid retained by the residues. The former is due to defective powdering, and may be controlled; with experience it is of infrequent occurrence. The retained tungstic acid does not appear to be within the control of the assayer, but proof of its presence is easy. Wolfram is not soluble in cold dilute caustic soda solution, whereas the retained tungstic acid is. Thus in ten recorded assays the quantity of tungstic acid (not including unattacked wolfram), expressed in percentage, extracted from the residues with cold dilute soda solution, was 0·05, nil, 0·55, 0·25, 0·7, 1·57, 1·1, 2·0, 1·1, 1·9, and 0·55 per cent., respectively.

Impurities frequently present in wolfram concentrates are silica and silicates, arsenic as mispickel, titanium, tantalum, niobium, and fluorine as fluorspar.

The presence of silicates and arsenates soluble in hydrochloric acid would give low results, some of the tungsten being dissolved and remaining soluble even in aqua regia. Fortunately such impurities seldom occur.

Arsenic as mispickel does not interfere with the assay; thus, a sample of nearly pure wolfram with no arsenic added gave 75·6 per cent., and with mispickel added, corresponding to 2·5 per cent. arsenic, assayed 75·65 per cent.

Titanium as ilmenite causes the retention of large amounts of tungstic acid in the residues when dissolving out with ammonia.

In an assay of alluvial wolfram concentrate, containing about 10 per cent. TIO_2

as ilmenite (see No. 5 in table), the amount retained by the residue and soluble in weak caustic soda solution was 3·6 per cent. The addition of 0·5 grm. of ilmenite to an assay of a clean wolfram concentrate caused a retention of 11 per cent. Tantalite and columbite do not affect the assay.

The difficulty experienced by some in the decomposition of wolfram has led to various methods being devised. All are somewhat similar in principle—viz., a preliminary ignition with some suitable material is given, the effect of which is to render the wolfram more easily attacked by acids. A brief account of some original work on these lines may be useful, if only to serve as a warning of the possible pitfalls attending the use of such methods.

One grm. of the wolfram is mixed with 0·6 to 0·7 grm. of ammonium chloride and 3·5 grms. of calcium carbonate; the mixture is heated in a platinum crucible (containing a layer of calcium carbonate at the bottom and top of the charge) for five minutes at a low heat, and then for five minutes at full bunsen heat. The fritted mass may be detached from the crucible when cold. It is sufficient then to boil with dilute or strong hydrochloric acid for a few minutes only, to effect complete decomposition. Unfortunately, the ease of attack is the only good feature of the method. Some of the bad points are—(1) The pronounced solubility of the tungsten in the acid liquors; (2) the presence of only small quantities of arsenic very much increases the solubility effect.

It is admissible as an alternative to determine the tungstic acid in the ammoniacal solution by the mercurous nitrate method. The author is indebted to Mr. J. J. Beringer for first acquaintance with the use of this method for the particular purpose.

The details as given below are based on experience.

II. "AQUA REGIA–MERCUROUS NITRATE" METHOD.

Proceed as described in the "aqua regia-ammonium tungstate" method to the sentence (p. 399) ending "wash with water, working so as to obtain as little tungstic acid on the paper as possible." To the beaker add about 15 c.c. distilled water and 5 c.c. of 10 per cent. caustic soda solution; cleanse the cover-glass with a few drops of very dilute solution, and wash into the beaker; run a few c.c. of dilute soda through the filter-paper, and wash with water, collecting filtrate and washings in beaker; rub down any deposit above the level of the liquid in the beaker with a rubber-tipped rod, raise to the boil, add 3 grms. ammonium nitrate, and continue to boil for a minute or so. Allow to settle; filter, and wash with weak ammonium nitrate solution, collecting filtrate and washings in a large-sized beaker. Dilute to about 200 c.c.; acidify with nitric acid first, then neutralise or make faintly alkaline with dilute ammonia. Add sufficient mercurous nitrate solution, and again neutralise with precipitated mercuric oxide; stir well; filter and wash by decantation; finally transfer all precipitate to the paper and complete the washing. Dry, burn, and weigh as tungstic acid. The residues should be again treated with acid, etc., for unattacked wolfram.

This is a useful alternative method to the ordinary aqua regia method, and gives results comparable with it (samples Nos. 1, 2, and 3, in the table). It is most advantageous for the assay of mixed tin-wolfram concentrates.

III. "SODA DIGESTION–MERCUROUS NITRATE" METHOD.

Digest 0·825 grm. of the finely powdered sample with 20 c.c. of a 25 per cent. solution of caustic soda in a porcelain basin on a water-bath for forty-five minutes; stir well at the commencement to prevent caking; dilute the extract with water, add a little sodium peroxide, transfer to a 250-c.c. flask, and dilute to 250 c.c. To 200 c.c. of a filtered portion (this will represent 0·5 grm. of the material) transferred to a large flask add nitric acid till acid, and then make alkaline with ammonia. Boil, filter, and wash into a suitable-sized beaker. Add to the filtrate containing the tungsten dilute nitric acid till faintly alkaline, then a solution of mercurous nitrate in slight excess; again neutralise with precipitated mercuric oxide; stir well; allow to subside, filter, and wash, first by decantation, and later when transferred to the paper. Dry, burn in platinum crucible, and weigh as tungstic acid. It is advisable to treat the tungstic acid with a little hydrofluoric acid, ignite, and again weigh. The loss, which is usually small and negligible, represents silica.

Fine grinding of the sample and thorough washing of the mercurous tungstate are both essential to good results. The method gives results comparable with the other methods (see Nos. 1, 4, 5, 7, 8, and 9, in the table).

Titanium as ilmenite, tantalite and arsenic as mispickel, and small quantities of arsenate, do not interfere. Large quantities need no consideration, since they do not occur in practice.

IV. "SODA FUSION–MERCUROUS NITRATE" METHOD.

Fuse 0·625 grm. of the finely powdered sample in a nickel dish with about 1 inch of stick soda and 3 grms. sodium peroxide, at first gently, then more strongly, until the fusion is complete. Pour the molten charge on to a clean sheet of nickel, and when cold transfer both dish and caustic mass to a beaker, and dissolve in water. If the solution is green, add a little peroxide of soda, transfer to a 250 c.c. flask, and make up to volume. Filter, and determine the tungsten in 200 c.c. as described in Method III.

Titanium, tantalum, and niobium, do not interfere. Arsenic, unless in excessive amount, is removed, or practically so, by the tin dissolved in the fusion to such an extent as to be too small to influence the assay.

In the table are set out assay results of a considerable number of samples, from which a comparison of the various methods may be made. It is impossible to make such a series of assays without developing a partiality for one or other method; of the four methods dealt with, the old aqua regia method (Method I.) has most to commend it. It gives comparable results on amounts varying from 1 to 2·5 grms., whereas the other methods are limited to quantities of about 0·5 grm. All four methods work well in the presence of scheelite.

Samples 9 to 28 inclusive are of interest, inasmuch as there are given both buyer's and seller's assays on a total quantity of about 250 tons, representing a transaction of considerably over £25,000. The buyer's average assay is 63·58 per cent., the seller's 63·56 per cent.

Table of Assay Results.

Sample Number.	Buyer's Assay.	Method I. "Aqua Regia-Ammonium Tungstate."	Method II. "Aqua Regia-Mercurous Nitrate."	Method III. "Soda Digestion-Mercurous Nitrate."	Method IV. "Alkaline Fusion-Mercurous Nitrate.
1	—	55·10	55·05	54·90	—
2	—	56·90	56·75	—	—
3	—	64·00	63·84	—	—
4	—	66·09	—	65·92	—
5	—	57·10	—	57·00	57·0
6	—	65·90	—	—	65·8
7	—	65·28	—	65·06	—
8	—	63·65	—	63·80	—
9	64·10	64·15	—	64·02	—
10	63·30	63·34			
11	63·95	63·90			
12	64·77	64·89			
13	63·55	64·00			
14	63·58	63·78			
15	64·10	64·00			
16	63·25	63·10			
17	63·03	62·70			
18	62·75	62·84			
19	63·00	62·65			
20	64·10	63·40			
21	63·80	63·85			
22	63·70	63·85			
23	63·80	63·85			
24	63·80	63·50			
25	64·00	63·80			
26	63·35	63·75			
27	62·75	63·00			
28	62·35	62·25			

Description of Samples.

1. Spanish wolfram recovered from waste products.
2. Cornish wolfram from lode material.

3, 4, and 6. Alluvial wolfram, locality unknown.

5. Titaniferous alluvial wolfram from Cornwall.

7. Spanish concentrate.

8 to 28 inclusive. Cornish wolfram from lode material.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Solubility of Carbon Dioxide in Beer. A. Findlay and B. Shen. (*Proc. Chem. Soc.*, 1911, **27**, 189.)—Previous experiments on the absorption of carbon dioxide by beer (Lange and Schultze, *Zeitsch. fur das ges. Brauwesen*, 1879, **2**, 369) have been interpreted as showing that beer dissolves more carbon dioxide than the corresponding water-alcohol solution, and the supposed increase has been attributed to adsorption by the colloids in the beer (Emslander and Freundlich, *Zeitsch. physikal. Chem.*, 1904, **49**, 317). These conclusions are not, however, in harmony with experiments carried out by Findlay and Creighton (*J. Chem. Soc.*, 1910, **97**, 536), and the authors now show that carbon dioxide is, as a matter of fact, less soluble in beer than in the corresponding water-alcohol solution, and that the contrary conclusion reached by Langer and Schultze must be attributed to their having used beer supersaturated with carbon dioxide.

Detection of Adulteration of Butter by Ewer's Method. C. Amberger. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 598-606.)—Ewer's method (ANALYST, 1910, **35**, 353) is stated to be capable of detecting the presence of 10 per cent. of cocoanut oil in butter, but it is shown by the results of experiments carried out by the author that this claim only holds good in the case of normal butter. The results obtained, particularly the "difference figure," are influenced by the period of lactation, and butter prepared from the milk of a cow at the end of the lactation period yields a "difference figure" which would point to the presence of more than 10 per cent. of cocoanut oil; these butters yielded Reichert-Meissl values of from 18·15 to 21·7, and were otherwise abnormal from an analytical point of view. Ewer's "difference figure" is also affected when the animals are fed on cocoanut oil cakes or beetroots, and the method may therefore, in special cases, yield results on which erroneous conclusions may be based. W. P. S.

Estimation of Cantharidine in Tincture and Oil of Cantharides. R. Gaze. (*Apoth. Zeit.*, 1911, **26**, 332-333; through *Chem. Zentralbl.*, 1911, I., 1657-1658.)—The author has obtained good results by the following procedure in the case of tinctures: 50 grms. of tincture, 25 grms. of water, and 1 c.c. of sodium carbonate solution (1 : 2), are evaporated together on the water-bath to dryness. The residue is taken up with 10 c.c. of water, acidified with 2 c.c. of 25 per cent. hydrochloric acid, and shaken out with 10 c.c. of chloroform which have previously been used to rinse out the flask. The chloroform is evaporated, and the residue, after standing for twelve hours, is extracted with 10 c.c., and then four times successively with 5 c.c., of light petroleum spirit. The petroleum washings are passed through a filter, and the residue and the filter are subsequently washed with 10 c.c. of water containing a trace of ammonium carbonate, and finally with pure water; the filter and residue are dried at 50° C. The product is dissolved in acetone, the solution is passed through the filter, the solvent is evaporated, and the cantharidine is dried at a temperature below 100° C. The average yield from 100 grms. of tincture prepared from 10 grms. of cantharides is 0·0684 grm., whilst the 10 grms. of cantharides contain 0·1079 grm. For the estimation of cantharidine in *Oleum Cantharidum* 20 grms. of the oil are shaken with 40 grms. of benzene, 1 c.c. of 25 per cent. hydrochloric acid, and 10 c.c. of water. A further 20 c.c. of water are then added, and 5 c.c. of sodium carbonate solution (1 : 2), and the mixture is again shaken for two minutes. The alkaline liquid is separated, and the benzene solution is again shaken with 20 c.c. of water and 5 c.c. of sodium carbonate; lastly, it is washed with 25 c.c. of water. The alkaline solution is evaporated to dryness on the water-bath, the residue is taken up with water, the solution is filtered, acidified, and shaken out with chloroform, the extract being treated as above. Twenty grms. of the oil yielded 0·0194 grm. of cantharidine. J. F. B.

Identification of Cocaine and Substitutes. F. J. Seiter and F. Enger. (*Amer. J. Pharm.*, 1911, **83**, 195-201.)—Cocaine salts react, under certain conditions, with solutions of gold chloride, platinum chloride, chromic acid, and potassium permanganate, to form micro-crystalline precipitates of definite and characteristic forms. Chlorine-water also serves as a useful reagent in distinguishing between cocaine and its substitutes.

The results obtained are shown in the following table:

Reagent.	Cocaine.	α-Eucaine.	β-Eucaine.	Stovaine.	Holocaine.	Acoine.	Euphthalmine.
$AuCl_3$ – 1 per cent. solution.	Fern-like crystals.	Twig-like crystals.	Amorphous precipitate.	Branched crystals.	Amorphous precipitate.	Amorphous precipitate.	No precipitate.
$PtCl_4$ – 1 per cent. solution.	Feathery crystals.	Fine needles.	Leaves, cubes, and rosettes.	No precipitate.	Small stars.	Amorphous precipitate.	No precipitate.
$KMnO_4$ – 1 per cent. solution.	Violet-red squares; slow reduction.	No crystals; slow reduction.	No crystals; slow reduction.	No crystals; slow reduction.	No crystals; immediate reduction.	No crystals; immediate reduction.	No crystals; immediate reduction.
H_2CrO_4 – 5 per cent. solution.	Fine needles; no reduction.	No crystals; no reduction.	No crystals; no reduction.	No crystals; no reduction.	No crystals; no reduction.	No crystals; brown precipitate and solution.	No precipitate.
Chlorine-water.	No precipitate.	Milky turbidity.	Dense turbidity.	Light turbidity.	Yellow turbidity.	Maroon precipitate and claret solution.	No precipitate.

The alkaloid, isolated in the usual ways, is converted into the hydrochloride, and made up to a 2 per cent. solution. Half a c.c. of this is treated with 5 drops of permanganate solution, and the crystals examined under the microscope. Other common alkaloids and the cocaine substitutes immediately or slowly reduce the solution without formation of crystals (*cf.* Hankin, ANALYST, 1911, 2).

A 1 per cent. solution of the alkaloidal substance in quantities of 0·5 c.c. is used for the platinum chloride and chromic acid tests, and 0·5 per cent. solution for the gold chloride test. Two drops, 2 drops, and 3 drops of the respective reagents are used. The precipitate at first formed with chromic acid is redissolved in hydrochloric acid. A. R. T.

A Casein Product. R. Windisch. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 613-615.)—Analyses are given showing the composition of a casein or curd product which is obtained in Hungary as a by-product during the preparation of sour cream. After the cream has been allowed to undergo lactic fermentation, the layer of cream is removed, and the remainder of the liquid is heated until the curd has coagulated into lumps. The latter are then collected and subjected to gentle pressure, the resulting product being used as a food. Analyses of 123 samples of the substance yielded the following average results: Water, 70·05 per cent.; dry solids, 29·95 per cent.; and ash, 0·70 per cent. The quantity of fat present varied from 0·42 to 15·55 per cent. More than one-half of the samples contained less than 20 per cent. of fat, calculated on the dry substance, the lowest fat-content of the dry substance being 1·50 per cent., and the highest 42·80 per cent. W. P. S.

Colour Reactions of Edible Fats and Oils. H. Serger. (*Chem. Zeit.*, 1911, **35**, 581-582, 602-603, 610-612.)—The author has collated the various tests which have been proposed for the purpose of identifying certain oils by means of colour reactions. The tests described include Welman's reaction, Bellier's reaction, Serger's reaction, Kreis's reaction, Wiedmann's reaction, Halphen's reaction, Becchi's reaction, Baudouin's reaction, etc., together with the colorations produced by the oils when the latter are treated with concentrated acids and mixtures of the same. The oils and fats dealt with are cotton-seed oil, sesame oil, olive oil, linseed oil, poppy oil, arachis oil, rape oil, almond oil, peach-kernel oil, cacao butter, cocoanut oil, lard, and beef fat. W. P. S.

Oil from the Seeds of Xanthophyllum lanceolatum. C. Grimme. (*Chem. Rev. Fett- u. Harz-Ind.*, 1911, **18**, 125-126.)—The seeds produced by *Xanthophyllum lanceolatum*, a tree belonging to the Polygalaceæ, yield a fat which is extensively used as food by the natives of Singapore. A sample of the seeds examined by the author yielded 39·17 per cent. of a fat with the following characteristics:

FAT.

Sp. Gr. at 50° C.	Melting-Point.	Solidification-Point.	$[N]_D$ 40° C.	Acid Value.	Saponification Value.	Iodine Value.	Hehner Value.	Glycerol.	Unsaponifiable Matter.
	° C.	° C.						Per Cent.	Per Cent.
0·9021	44·0	41·5	1·4549	52·06	189·5	38·9	95·85	6·41	1·37

FATTY ACIDS.

Melting-Point.	Solidification-Point.	$[N]_D$ 55° C.	Iodine Value.	Mean Molecular Weight.	Neutralisation Value.
° C.	° C.				
55	53·5	1·4424	38·2	268·3	207·2

The fat was not toxic, but the press-cake contained a poisonous saponin, which would render it unsuitable for a feeding-stuff. C. A. M.

Examination of Ether for Anæsthetic and Analytical Purposes. C. Baskerville and W. A. Hamor. (*J. Ind. Eng. Chem.*, 1911, **3**, 378-398.)—The authors have made trial of all the published tests, and as a result they have drawn up a scheme for the examination of ether with particular reference to the detection of avoidable impurities. The scheme includes twelve tests, of which the following, for the detection of peroxides, is new :

The reagent is a 10 per cent. solution of cadmium potassium iodide, which is preferred to potassium iodide because strong solutions of the latter decompose so readily in sunlight. Of this reagent, 2 c.c. is added to 10 c.c. of the ether to be tested, and the mixture is well shaken and allowed to stand for an hour. There should be no visible liberation of iodine within that time. The limit of sensibility of the test is about 0·0025 per cent. of hydrogen peroxide. G. C. J.

Influence of Certain Vegetable Proteins on Wheat Gluten. B. von Fenyvessy. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 658-662.)—Results of experiments are recorded regarding the effect of the addition of different vegetable proteins to wheat flour, the main object being to ascertain the influence of these substances on the physical properties of the gluten of the flour. The addition of wheat gliadin, in quantities varying from 5 to 20 per cent. of the weight of the flour, increased the weight and baking qualities of the gluten ; glutenin also increased the weight of the gluten, but diminished its baking quality. These experiments confirm the statement that the quality of a gluten depends on its composition. Gliadin prepared from rye flour acted in the same way as that obtained from wheat. The fact that rye flour itself does not yield a coherent cake of gluten is due, in the author's opinion, to the presence of substances other than glutenin in this flour. Hordein prepared from barley decreases the baking quality of wheat flour, whilst zëin obtained from maize has no appreciable effect. W. P. S.

Analytical Examination of Linseed Oil. H. R. Jensen. (*Pharm. J.*, 1911, **86**, 839-840.)—The author outlines the following scheme for the examination of linseed oils : Five grms. of oil are saponified for one hour with 6 per cent. alcoholic potash, and the unsaponifiable matter examined for rosin and mineral oils. The fatty acids are thrown out with hydrochloric acid (sp. gr. 1·05) and thoroughly extracted with ether ; the mixed ether extracts are washed with three quantities of 6 c.c. each of water, and the volume made up to 100 c.c. with anhydrous ether. A yield of fatty acids equal to about 93·6 per cent. of the weight of oil taken is generally obtained. To 25 c.c. of this ether extract, contained in a rubber-stoppered flask, are added 15 c.c. ether and 3 c.c. glacial acetic acid; the mixture is cooled in ice-water, and 0·5 to 0·8 c.c. of bromine added, in drops, till in excess. After standing for two to three hours at the same temperature, the precipitate is collected on two interleaved counterpoised filter-papers, the flask and precipitate being washed with six quantities of 8 c.c. each of cooled ether (sp. gr. 0·720), to remove free bromine and lower soluble compounds, then dried at 95° C. and weighed. Too high a melting-point, with yield too high in proportion to iodine value, suggests adulteration with fish oils; too low a yield, an admixture of vegetable and mineral oils. To obtain the actual weight of acids in the ether extract, 25 c.c. are rapidly evaporated in a beaker at 100° C., with the addition of 5·5 c.c. absolute alcohol, the lowest weight obtained being taken as the amount of fatty acids in solution. The melting-point and refractive index of this residue are determined. The ether solution may be used for determining the iodine value, 25 c.c. being diluted to 250 c.c. with any inert solvent, and 25 c.c. solution taken, using 20 c.c. Wijs or Hanŭs solution and 10 c.c. carbon tetrachloride, with two hours' contact. The ether present renders necessary a correction, a 5·6 per cent. reduction of the observed iodine value being made, or, better, avoided by the use of tetrachloride only. The same solution of fatty acids is employed for determining the neutralisation value. The Liebermann reaction cannot be generally applied to the detection of rosin and rosin oils in linseed oil, as the latter contain sitosterol, giving a bluish-violet colour, reddening on standing. The following figures were obtained by the above processes:

	Standard Limits for Linseed Oil.	Laboratory-pressed River Plate Oil, Settled and Filtered.	An Oil apparently Genuine.	An Admittedly Adulterated Oil (Small Quantity Fish Oil present).
Colour	—	pale	pale	medium
Sp. gr. at 15·5° C.	0·9305 to 0·937	0·9319	0·931	0·932
[N]$_D$ 15° C.	1·482 to 1·485	1·482	1·4825	1·4825
Iodine value (Hanŭs)	172 to 200	175·6	176	179
Saponification value	187 to 195	191·5	192	193·9
Unsaponifiable matter, per cent.	0·5 to 1·8	0·88	0·56	1·45
Acid value	0·8 to 3·0	1·3	1·0	2·4
Ether-insoluble hexabromo-glycerides ...	23 to 26 per cent. (−38%=190 I.V.)	44	44·9	47·5
Ether-insoluble hexabromo-glycerides, melting-point °C.	—	118 to 141	124 to 142·5	125 to 143
Liebermann-Burchard reaction	—	faint	faint	distinct
$H_2SO_4-CS_2$ liver oil reaction	negative	negative	negative	faint
Fatty acids + unsaponifiable matter, per cent.	94·8 to 95·5	94·5	94·1	95·5
Mixed fatty acids, iodine value	179 to 210	180·5	183·3	188·3
,, ,, melting-point °C. ...	17 to 22	22	—	21·5
,, ,, [N]$_D$ 20° C.	[1·4546, 60°]	1·472	—	1·472
,, ,, Neutralisation value ...	196 to 199	209·9	205·6	203
,, ,, Mean molecular weight	283 to 307	266·8	272·4	275·8
Ether-insoluble hexabromides from fatty acids	29 to 31 per cent. (−41·9 %=190 I.V.)	36·6	34·9	35·5
Ether-insoluble hexabromides from fatty acids, melting-point °C.	175 to 180	176 to 178·5	179 to 180	177 to 181·5

A. R. T.

Estimation of Glycyrrhizin and Sugars in Liquorice Powder and Extract. E. Eriksson. (*Arch. Pharm.*, 1911, **249**, 144-160.)—The method proposed is based on the fact that glycyrrhizin is decomposed by hydrolysis into one molecule of glycyrrhetic acid and two molecules of glycuronic acid, and since the latter reduces Fehling's solution, the glycyrrhizin, dextrose, and sucrose may be estimated at the same time. The solution of the liquorice powder is prepared by mixing 10 grms. with an equal quantity of glass powder and a little water in a mortar, and allowing it to stand for some hours, after which the mixture is treated with about 50 c.c. of water, containing three to four drops of alkali solution per 100 c.c. (to convert any free glycyrrhizic acid into its soluble alkali salt), and allowed to stand overnight. The liquid is then made to percolate slowly, its place being continually renewed by fresh alkaline water, until, after about 100 c.c. have been collected, the drops have no taste. The percolate is made up to 200 c.c., 40 c.c. mixed with an equal volume of 90 per cent. alcohol, and heated on the water-bath to precipitate mucilaginous substances, the alcohol evaporated as rapidly as possible, and the liquid filtered. The cold filtrate and washings are treated with 30 c.c. of Fehling's solution and allowed to stand in the cold for twelve to thirteen hours, after which the reduced cuprous oxide is collected, reduced to metallic copper, and the corresponding amount of dextrose calculated. The filtrate is next boiled for three minutes with 30 c.c. of Fehling's solution, then diluted with half its volume of water, and the cuprous oxide collected in an Allihn's tube, and weighed as metallic copper. The result gives the quantity of sugar (sucrose), estimated as hexose, which reduces Fehling's solution on boiling. The filtrate, which now contains only the glycyrrhizin, is treated little by little with 25 per cent. sulphuric acid until no further precipitation occurs. The precipitated glycyrrhizic acid is allowed to stand for two to three hours, then separated, washed carefully with 5 per cent. sulphuric acid, and converted into its potassium salt by treatment with 5 per cent. potassium hydroxide solution. The alkaline solution is filtered and boiled for fifteen hours with Fehling's solution under a reflux condenser. The amount of glycyrrhizic acid may then be calculated from the apparent amount of dextrose by means of the equation $360 : 896 = 50{\cdot}9 : x\ (= 1{\cdot}239)$.

A similar course is followed in the analysis of liquorice extract, and the glycyrrhizin may be estimated prior to the estimation of the dextrose and sucrose in other portions of the liquid. The following results were thus obtained in the analysis of commercial samples of the air-dried powders and extracts:

	Liquorice Powders.						Liquorice Extracts.						
	Italian.	Spanish "Tortosa."	Russian.	Russian.	Russian.	Fresh Root from Atri.	Cassano.	Parone Compagna.	S. Franca.	Corrigliano.	Barraco.	Atri.	Atri, prepared from Fresh Root.
Dextrose, per cent.	1·39—1·43	1·28	—	trace	3·80	—	6·30	3·79	2·70	7·82	15·20	5·90	4·50
Sucrose, per cent.	2·4 —2·57	3·20	6·48	6·50	6·25	2·60	11·80	4·52	8·17	9·06	11·90	12·48	13·60
Glycyrrhizin, per cent. ..	6·65—7·10	6·49	7·70	8·15	7·33	6·72	16·45	14·22	23·90	12·10	11·59	10·20	9·85

If the total amount of glycyrrhizin and sugars in the fresh root (9·12 per cent.) is compared with that in the extract made by the author from that root (27·95 per cent.), the proportion is approximately as 1 : 3. Hence it would seem that there must have been a transformation of some of these substances into one another in the concentration of the extract. C. A. M.

Analysis of Liquorice Juice. E. J. Parry. (*Chem. and Drug.*, 1911, 133; through *J. Soc. Chem. Ind.*, 1911, 30, 647.)—Since the publication of the papers of the author (*Chem. and Drug.*, January, 1910, p. 21), Eriksson (see preceding abstract), and Telle (*Ann. Falsif.*, 1911, 4, 3), a greater amount of reliable information in regard to the composition and analysis of liquorice juice is available than was the case before, and it should now be possible for a definite opinion to be expressed as to the purity of a given sample. The determinations recommended are: Moisture, mineral matter, glycyrrhizin, invert sugars, uninverted sugars, matter insoluble in water, matter insoluble in dilute ammonia, alcohol precipitate (see below); and microscopic examination of the matter insoluble in water and of that insoluble in ammonia. Adulteration of liquorice juice is practised to an enormous extent, and in France steps are being taken to establish standards of purity. The author suggests 9 per cent. of glycyrrhizin as a minimum for normally prepared edible liquorice juices; Spanish juices, which are much richer in glycyrrhizin, have too little "body" to be used alone. With regard to the sugars, the methods of Eriksson for the separate determination of invert sugars and sucrose are open to criticism; moreover, as liquorice root contains an enzyme, the relative proportions of inverted and uninverted sugars in the juice will vary greatly according to the method of preparation. The figures obtained by the author and by Eriksson indicate that a normal edible juice contains not more than 18 per cent. of total sugars. In order to distinguish between natural and added starch in liquorice juice, the sample should be powdered, extracted with water, and the residue extracted with 3 per cent. ammonia solution. The residue should not exceed 6 per cent., and should be compared microscopically with the starch present naturally in liquorice root. When alcohol is added to an aqueous extract of the juice until the alcohol concentration is 70 per cent., a precipitate is produced; according to Telle, this does not exceed 16·5 per cent. in the case of pure juices (except in some doubtful Levant and Spanish juices), whereas it is much higher in the presence of gum, gelatin, and commercial glucose containing dextrin.

Preparation of Madeira and its Composition. F. da Silva. (*Ann. Falsif.*, 1911, 4, 57-65.)—The author describes the usual process followed in the manufacture of Madeira, and gives the results of the analysis of a large number of samples. His general conclusions are as follows: The alcohol amounts on the average to 20 per cent., with a maximum of 23·30 and a minimum of 16·60 per cent. The extract shows an average of 78 grms., with a maximum of 185 and a minimum of 31·6; after deducting the unfermented or added reducing sugars, it averages 30 grms., with a maximum of 82 and a minimum of 12·7 grms. The proportion of tannin estimated by the zinc acetate method is only small—the maximum is 1·05, the minimum 0·07, mean 0·15. The total acidity is generally high, averaging 5 grms. per litre. The sum of the alcohol and acid shows an average of 25, maximum 29, minimum 20; these values are only quoted incidentally, and cannot be compared with those found for ordinary wines. Madeiras are not "dry," but contain a more or less considerable quantity of uninverted sugar. Tannin is present in all Madeiras, the Verdelho wine generally containing more than the Serical wine. J. F. B.

Higher Alcohols occurring in Jamaica Rum. T. von Fellenberg. (*Mitt. Lebensmittelunters. u. Hyg.*, 1911, **1**, 352-357.)—The higher alcohols of Jamaica rum consist chiefly of normal butyl alcohol; small quantities of propyl alcohol are also present, together with traces of isobutyl and amyl alcohols.

Green Colour of Rapeseed Cakes. L. Bussard. (*Ann. Falsif.*, 1911, **4**, 75-77.)—Indian rapeseed cakes are frequently treated with from 0·75 to 1 per cent. of sodium carbonate in order to give them the green colour shown by the European product.

Estimation of Salicylic Acid in Fruit Juices. P. Vierhout. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 664-666.)—The following procedure is recommended for the estimation of salicylic acid in fruit juices, the main feature of the process being the prevention of a persistent emulsion when the juice is shaken with the solvent. For this purpose a considerable quantity of alcohol is added in order to precipitate the pectinous substances present, and the salicylic acid is extracted by means of petroleum spirit, the author having proved that this solvent readily extracts salicylic acid from a dilute alcoholic solution. Fifty c.c. of the juice are acidified with 15 drops of concentrated sulphuric acid, and shaken with 50 c.c. of petroleum spirit (b.p., 50° C.); 25 c.c. of 96 per cent. alcohol are then added, the mixture is again shaken, and, when separation has taken place, the petroleum spirit layer is siphoned off into a graduated cylinder. The alcoholic portion is once more shaken with 50 c.c. of petroleum spirit, after the addition of a further quantity of 25 c.c. of alcohol, and the whole mixture is transferred to the measuring cylinder, the contents of which are diluted with a mixture of 5 parts of petroleum spirit and 2·5 parts of alcohol to a volume of 200 c.c. After shaking, 50 c.c. of the petroleum spirit (equal to 12·5 c.c. of the juice) are transferred to a small corked flask containing a few grms. of anhydrous sodium sulphate. At the end of two hours the solution is filtered through a dry filter into a flask containing 50 c.c. of $\frac{N}{10}$ sodium hydroxide solution, the flask and the sodium sulphate are rinsed with the alcohol-petroleum spirit mixture, and, after the petroleum spirit has been evaporated from the alkaline filtrate, the excess of sodium hydroxide is titrated. A simple calculation then gives the quantity of salicylic acid present. W. P. S.

Tobacco Extracts and their Value. J. Schröder. (*Zeitsch. anal. Chim.*, 1911, **50**, 433-435.)—Large quantities of tobacco-juice are imported into South America to combat sheep-scab and ticks, the chief exporting countries being

Austria, Italy, and the United States. The author considers that payment for this product should be based on the unit of nicotine. At present the price is never less than forty nor more than fifty centésimos per kilo, but the product may contain anything from 1·5 to 11 per cent. of nicotine. The author estimates nicotine in extracts as follows: The extract (10 grms.) is mixed with so much dry sand that a dry powder results. This is slightly moistened with alcoholic potash, placed in a thimble, and extracted with ether for five hours. The extract is cautiously evaporated to two-thirds its bulk on the water-bath, and to the residue 200 c.c. of 1 per cent. aqueous caustic soda are added, and the mixture distilled in a current of steam until the distillate amounts to 500 c.c., the residue in the flask at the end of the operation being 60 to 80 c.c. The nicotine in the distillate is then estimated by titration with $\frac{N}{10}$ sulphuric acid. G. C. J.

Estimation of Tannin in Wines. P. Malvezin. (*Bull. Soc. Chim.*, 1911, 9, 620-621.)—Ten c.c. of the wine are boiled for five minutes with 10 c.c. of a solution of zinc acetate, the mixture then cooled, and treated with 25 c.c. of boiling water, and the precipitate collected and washed with about 100 c.c. of boiling water. The zinc acetate solution is prepared by dissolving 10 grms. of precipitated zinc oxide in the requisite quantity of acetic acid, adding 80 c.c. of ammonia solution, and diluting the liquid to a litre. The precipitate obtained with this reagent is dissolved off the filter by means of dilute sulphuric acid (2·5 c.c. of acid to 100 c.c. of water), the filter-paper washed with 20 c.c. of water, the solution and washings heated to 60° to 70° C., and titrated with $\frac{N}{10}$ potassium permanganate solution, which is added 5 drops at a time until the coloration persists for two minutes and the liquid has a turbid appearance in transmitted light. The number of c.c. used, multiplied by 0·116, gives the amount of gallotannic acid in grms. per litre. The method is stated to give very accurate results. C. A. M.

Detection of Tannin in Solution. G. Grasser. (*Collegium*, 1911, 46-52.)—A method of detecting tannin is based upon the fact that it absorbs iodine, and that the latter will therefore give no coloration with starch until present in excess. Thus, on treating a solution of tannin with a few drops of a dilute solution of iodine and adding starch solution, the presence of the tannin is indicated by the absence of the usual coloration. Only in the case of pine-bark was any coloration that would mask this test observed. Comparative tests with different reagents applied to solutions of gallotannic acid, mangrove infusion, and tannin solution containing much non-tannin substance, showed that in each case $\frac{N}{50}$ iodine solution was the most sensitive, being capable of detecting 0·0001 per cent. of gallotannic acid, 0·01 per cent. of tannin in mangrove infusion, and 0·0034 per cent. of tannin in the liquor rich in non-tannin substances. Zinc acetate in ammoniacal solution detected 0·0005 per cent. of gallotannic acid, but is less reliable than iodine, owing to the fact that it probably precipitates substances other than tannin. The best results were obtained with a solution containing 1 per cent. of gelatin and 10 per cent. of sodium chloride, and in the author's opinion this is the most reliable reagent. Other reagents tried (5 per cent. solutions of tartar emetic with and without the addition of other salts) were less sensitive. Experiments showed that the various tannins do not precipitate one another. C. A. M.

Detection of Turpentine Adulteration. (*U.S. Department of Agriculture, Bureau of Chemistry Bulletin*—Oil, Paint, and Drug Report, April 17, 1911.)—The following are suggested as specifications for three standard grades of turpentine: Standard or No. 1 turpentine should have a sp. gr. at 20° C. of from 0·862 to 0·870; $[\mathrm{N}]_{\mathrm{D}20^\circ\mathrm{C.}}$ 14·68 to 1·476; 95 per cent. should distil below 170° C., and a layer of not less than 200 mm. should be required to equal in colour the Lovibond yellow glass No. 1. On polymerisation with 38 N sulphuric acid the residue should not exceed 1 per cent., should be reddish in colour and viscous, and its $[\mathrm{N}]_{\mathrm{D}20^\circ\mathrm{C.}}$ should be from 1·500 to 1·520. An unadulterated turpentine which does not agree with these requirements may properly be regarded as not of standard or No. 1 quality.

Second quality or No. 2 turpentine should have a sp. gr. at 20° C. of from 0·862 to 0·875; $[\mathrm{N}]_{\mathrm{D}20^\circ\mathrm{C.}}$ 1·468 to 1·480; 90 per cent. should distil below 170° C., and a depth of not less than 100 mm. should be required to equal the Lovibond yellow glass No. 1. The polymerisation residue must not exceed 1 per cent., and must have $[\mathrm{N}]_{\mathrm{D}20^\circ\mathrm{C.}}$ of not less than 1·50.

Third quality or No. 3 turpentine should have a sp. gr. at 20° C. of from 0·865 to 0·880; $[\mathrm{N}]_{\mathrm{D}20^\circ\mathrm{C.}}$ 1·468 to 1·485; 60 per cent. should distil below 170° C., and a depth of not less than 50 mm. should be required to equal the Lovibond yellow glass No. 1. The polymerisation residue must not exceed 1 per cent., and must have $[\mathrm{N}]_{\mathrm{D}20^\circ\mathrm{C.}}$ of not less than 1·500.

The following are the tests for adulteration:

"Straight" wood turpentines are readily distinguished from gum turpentines by their odour, or when they have been very carefully refined, by the odour of the first fraction or of the residue from fractional distillation. One or both of these portions have the peculiar "sawmill smell," and the residue has a camphoric and somewhat nauseating odour characteristic of wood turpentine, which is quite different from the mild, sweet fragrance of gum turpentine.

Destructively distilled wood turpentine and also rosin spirits are more readily distinguished from gum spirits by their odour than is wood turpentine prepared by steam distillation, and they also give distinguishing colour reactions when mixed with sulphurous acid, as suggested by Herzfeld, or with hydrochloric acid (the presence of tin or zinc is not necessary), as suggested by Grimaldi. When steam-distilled wood turpentine has been carefully refined so that 90 to 95 per cent. of it distils at from 155° to 170° C., both of these colour tests are practically useless.

Turpentine adulterated with more than 10 to 20 per cent. of coal-tar oils, or of gasoline or kerosene which have not been deodorised, may usually be readily detected by the characteristic odour of the mixture. The odour of rosin spirits, while quite distinctive, is difficult to detect in mixtures with turpentine. The presence of petroleum oils is also indicated by bubbles, or "beads," persisting for a few moments on the surface of the turpentine shaken in a partly filled bottle.

The presence of more than about 10 per cent. of kerosene or similar mineral oils is readily detected by the spot which a few drops of the sample placed on white paper leaves on drying. Gasoline and other light mineral oils do not leave this spot.

It is possible so to adulterate turpentine that neither the sp. gr., refractive index, or flash-point, is materially altered. Such careful adulteration is rare, but as adulterants cannot invariably be detected by making these determinations, and, further, as mineral oil is the most probable adulterant, it is more rational to at once polymerise the suspected sample, note the volume, colour, and consistency of the unpolymerised residue, and determine its refractive index.

If rosin spirit is present in notable quantities, it may be detected by the amount of residue on polymerisation, in conjunction with lowered initial boiling-point and the deep coloration produced by mixing portions of the original turpentine of the first fraction with sulphurous acid and with hydrochloric acid.

If the initial distilling temperature of the turpentine is less than 154° C., and the original sample gives a reddish colour with sulphurous acid and a greenish-red with hydrochloric acid, and the residue from polymerisation is less than 5 per cent., it is probable that the sample is adulterated with rosin spirits. An odour of rosin spirits in the sample, or in any fraction, would be confirmatory, and all tests should be repeated on the first fraction obtained with a fractionating column. At best the detection of small amounts of rosin spirits is very difficult, and the procedure outlined may often fail to detect its presence.

The presence of marked quantities of coal-tar oils, benzol, toluol, and xylol, is detected by the odour, the lower initial distilling temperature—*i.e.*, between 80° and 155° C.—and by further examination of the first fractions which distil below 155° C. The rise of temperature on shaking during polymerisation is also indicative of the nature of the sample. Coal-tar and petroleum oils do not raise the temperature more than 35° or 40° C. when mixed with sulphuric acid. When the temperature of turpentine mixed with 38 N sulphuric acid does not rise above 50° or 60° C., mineral oil, coal-tar oil, or some other oil than turpentine, is present in large quantities. The older the sample of turpentine, the greater, or at least the more rapid, the rise of the temperature of the mixture.

Non-volatile oils are easily detected by a distillation test.

Estimation of Pyridine in Vinegar. H. Fincke. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 655-658.)—Vinegar manufactured from denatured brandy (which in Germany contains about 0·45 per cent. of pyridine) contains a certain quantity of pyridine, but this quantity is not always proportional to the amount contained in the spirit, as the latter may have been mixed with other alcoholic liquids before the acetic fermentation. In order to estimate pyridine in vinegar, the author recommends a method proposed by Houghton (ANALYST, 1910, **35**, 32), which allows pyridine to be estimated in the presence of ammonia. Five hundred c.c. of the vinegar are acidified with sulphuric acid, and evaporated to a volume of about 40 c.c.; the residual liquid is then transferred to a flask, rendered alkaline, and distilled, the distillate being collected in a receiver containing hydrochloric acid. The acid distillate is transferred to a flask, rendered alkaline, and treated with sodium hypobromite solution in excess in order to decompose the ammonia. The contents of the flask are next distilled and the distillate collected in a measured quantity of $\frac{N}{10}$ hydrochloric acid. When the distillation is completed, the excess of the acid is titrated, using methyl orange as indicator. As a control, the neutralised solution may be titrated with $\frac{N}{10}$ sodium hydroxide solution after the addition of phen[illegible]phthaleïn, the alkali thus used being equivalent to the quantity of pyridine present. The amount of acid used in the first titration should be equal to the amount of alkali employed in the second titration. It is shown that the method yields trustworthy results. W. P. S.

Turpentine Oil from India. (*Bull. Imp. Institute*, 1911, **9**, 8-10.)—The oils are stated to have been prepared from the oleo-resin of the "chir" pine (*Pinus longifolia*), and gave the following results on examination:

			Commercial Russian Oils.	
	A.	B.	(1)	(2)
Sp. gr. at 15° C.	0·871	0·868	0·866	0·884
Optical rotation in 100-mm. tube	-0·75°	-2·17°	+14·5°	+16·3°
Fraction boiling at (per cent. by volume):				
145° to 160° C.	—	—	4	2
160° „ 165° C.	—	—	12	16
165° C. or below	1	1	—	—
165° to 170° C.	54	55	43	36
170° „ 175° C.	25	28	20	18
175° 180° C.	12	9	11	7
180° 185° C.			3	4
185° 190° C.			2	2
190° 195° C.			—	—
Residue	7	6	—	—

The No. I. Russian oil was also found to contain some petroleum.

The figures show the Indian oils to resemble Russian turpentine oils more closely than American oil. The Indian oils are more constant in composition than the Russian oils, which they could replace commercially in the cheaper grades of varnish and for use in black lacquers and disinfectant preparations. A. R. T.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

New Indicator for the Estimation of "Titratable" Alkali in Blood. P. Bernhardt. (*Wien. klin. Wochensch.*, 1911, **24**, 606-607; through *Chem. Zentralbl.*, 1911, I., 1763.)—The indicator consists of 2 volumes of a 1 per cent. aqueous solution of alizarinsulphazide and 1 volume of a 1 per cent. aqueous solution of Grübler's indigocarmine; the mixture, when the reaction is neutral or acid, possesses an intense dark green colour, which changes to a reddish-violet when there is the slightest excess of alkali. The estimation of the alkali in blood is performed by Engel's method, using $\frac{N}{75}$ tartaric acid solution and a special alkalimeter. J. F. B.

Coagulating-Points of Egg Serum and Milk Albumins, and of Serum Globulin, in Ammonium Sulphate Solutions. K. Micko. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 646-654.)—The author has shown previously (ANALYST, 1911, 13) that meat and egg albumins coagulate at different temperatures, and now shows that the method described not only allows the various albumins to be distinguished from each other, but also aids in their identification. The coagulating-point is taken to be that point at which a finely-divided flocculent precipitate is formed, and the determination is made with a solution of the albumin one-half saturated with ammonium sulphate; in the case of globulin, which is precipitated by this concentration of ammonium sulphate, the solution must be one-quarter saturated with the salt. The following coagulating points are recorded: Egg albumin, 71° to 72° C.; milk albumin, 76° to 78° C.; serum albumin, 74° to 76° C. When the albumins have been once precipitated with ammonium sulphate in the cold, their coagulating points are affected; egg albumin thus treated coagulates, under the above conditions, at 68° to 71° C.; milk albumin, 46·5° to 56·5° C.; serum albumin, 71° to 73° C.; and globulin, 66·5° to 74° C. A second precipitation with ammonium sulphate does not materially affect the coagulating points. W. P. S.

Influence of Viscosity on the Action of Invertase according to the Concentration of Saccharose. P. Achalme and M. Bresson. (*Comptes rend.*, 1911, **152**, 1420-1422.)—Opinions have been divided as to the influence of the concentration of sugar on the velocity of the action of invertase. O'Sullivan and Thompson concluded that the action of invertase, other things being equal, is proportional to the quantity of sugar present in the liquid, and that it increases or decreases with it. Duclaux, on the other hand, held that a given quantity of invertase produces an effect which is always the same without regard to the quantity of sugar present, and that it acts as a constant force which in a given time can only effect a definite amount of work. Other authors have found that in dilute solutions the inverting action is practically proportional to the quantity of sugar, but that with stronger solutions, starting from $\frac{N}{20}$ or 1·71 per cent., the velocity of inversion appears to be independent of the concentration of the sugar; lastly, for concentrations of 35 per cent. and over, the speed of inversion decreases as the concentration of sugar is increased. The present authors have verified this and find an explanation in the variations of the viscosity of the sugar solution according to its concentration. If, instead of calculating the absolute quantity of sugar inverted in a given time, the time be calculated which is necessary for the inversion of a given proportion—say 25 per cent.—of the original quantity of sugar present, a curve is obtained which is nearly horizontal for low concentrations and nearly vertical for high concentrations of sugar. At the same time a curve illustrating the increase of viscosity with concentration of sugar is found to run practically parallel with this time curve, the viscosity increasing only slowly at low concentrations and very rapidly at high concentrations. If the absolute quantity of sugar inverted in a given time be considered, it will be seen that the retardation due to the increase in viscosity acts in opposition to the acceleration due to the increase in concentration. For low concentrations the retardation is small, and allows the influence of mass action to be perceived; at moderate concentrations the two opposing influences are balanced; whilst at high concentrations the retardation due to viscosity increases faster than the acceleration due to concentration and becomes predominant.

J. F. B.

Estimation of Fat in Pathological Urine. S. Kakiuchi. (*Biochem. Zeitsch.*, 1911, **32**, 137-144; through *Chem. Zentralbl.*, 1911, I., 1722.)—Fifty c.c. of urine are heated in a beaker of about 200 c.c. capacity, with 14 c.c. of sodium hydroxide solution (sp. gr. 1·5) on the boiling water-bath for two hours, the beaker being covered with a watch-glass. After cooling, the contents are placed in a separating funnel of 500 c.c. capacity, treated with 30 c.c. of concentrated hydrochloric acid, and cooled. The liquid is then shaken with 70 c.c. of ether; the ethereal extract is separated, and the treatment is repeated with a fresh quantity of ether. The residue, after evaporation of the ether, is dried for two hours at 50° C., and then dissolved in light petroleum spirit. After standing for an hour, the petroleum extract is filtered through a layer of asbestos and the petroleum is evaporated. The dish containing the extract-residue is heated in a vacuum apparatus for three hours under a pressure of 30 to 40 mm. at the temperature of the boiling water-bath, and is weighed, after drying in a vacuum desiccator. Under these conditions, normal urine yielded only 0·0003 grm. of residue, whilst in a sample of chyluric urine, 0·423 grm. of residue per 100 grms. of urine was obtained, having the properties of a fatty acid of high molecular weight. J. F. B.

Hippuric Acid as a Cause of the Failure of the Spectroscopic Test for Hæmoglobin in Urine. F. A. McDermott. (*J. Amer. Chem. Soc.*, 1911, **33**, 992-995.)—Solutions of hippuric acid of a concentration such as may occur in urine are distinctly acid to litmus, and have the power of destroying hæmoglobin, or at least of rendering it inactive spectroscopically. If 2 drops of blood be added to 25 c.c. of a saturated solution of hippuric acid—a very dilute solution owing to the low solubility—hæmoglobin cannot be detected spectroscopically after five minutes, at the end of which time the colour of the solution will have changed from the pinkish tint of a dilute blood-solution to that of a strong solution of ferric alum. Since sodium hydroxide destroys the spectrum of hæmoglobin almost as fast as strong acids do, whereas ammonia has no such action, it is suggested that any sample o urine which must be preserved for spectroscopic examination should be neutralised, or rendered slightly alkaline with ammonia, at the earliest possible moment.

G. C. J.

ORGANIC ANALYSIS.

Detection and Estimation of Small Quantities of Acetic Anhydride in Acetic Acid. M. G. Edwards and K. J. P. Orton. (*J. Chem. Soc.*, 1911, **99**, 1181-1185.)—The authors base a method upon the fact that 2-4-dichloroaniline reacts rapidly with acetic anhydride, but not with acetic acid, at the ordinary temperature with formation of an anilide, readily separable and convertible into a chloroamine, and the amount of which can be determined.

Two grms. or more of 2-4-dichloroaniline are added to 100 c.c. of the acetic acid to be examined, and the mixture kept at 16° C. in a water-bath overnight. The reaction is rapid, but considerable time is required for the last traces of anhydride to disappear. The whole is diluted with water to 20 per cent. acetic acid, and the solution extracted with chloroform, 10 c.c., 7 c.c., and 5 c.c., of solvent being successively used for each 100 c.c. of the liquid. The aqueous solution is finally washed twice with 1 c.c. of chloroform. The whole of the anilide, together with part of the aniline, is thus removed, and the unchanged aniline is extracted from the chloroform by shaking with 10 per cent. hydrochloric acid, 160 to 200 c.c. being used for each grm. of aniline added. The acid liquid is finally washed twice with 1 c.c. of chloroform. The mixed chloroform extracts are added to an equal volume of glacial acetic acid, and dilute bleaching-powder solution slowly added, the amount of bleaching-powder solution being three to five times that required to convert all the anilide into chloroamine, and in volume equal to the glacial acetic acid, which is then diluted to 50 per cent. strength. The reversible reaction—

$$\mathrm{Ar.NHAc + Cl_2 \rightleftharpoons ArNClAc + HCl}$$

is then nearly complete.

Sufficient $\frac{N}{20}$ bleaching-powder solution is now added to dilute the acetic acid to 20 per cent. Thus, if 30 c.c. of glacial acid were first added to the chloroform solution, then (1) 30 c.c. bleaching-powder, and (2) 90 c.c. $\frac{N}{20}$ bleaching-powder solution should be added. Any red colour here shows the incomplete removal of aniline. The chloroform is now shaken with 30 to 40 c.c. of $\frac{N}{20}$ bleaching-powder solution to free it from acetic acid, and the solvent evaporated in a current of dust-free air at a temperature not exceeding 25° C. The orange-yellow coloured residue is dissolved in 5 to 10 c.c. of chloroform and treated with 5 per cent. potassium iodide and a little acetic acid, and the freed iodine titrated with $\frac{N}{20}$ thiosulphate. The results are satisfactory. A. R. T.

Proportion of Asphalt insoluble in Alcohol-Ether in Mineral Lubricating Oils. H. Loebell. (*Petroleum*, 1911, 6, 774-775; through *Chem. Zentralbl.*, 1911, I., 1722-1723.)—For the lubricating value of mineral oils the percentage of asphalt is not by itself a sufficient measure, a determination of the consistence is also an important factor. The author recommends the following modification of Holde's alcohol-ether method, in order to obtain a more rapid drying of the oil and asphalt: About 5 grms. of the oil are dissolved in 140 c.c. of ether, and precipitated by 70 c.c. of alcohol; after standing for five hours at 15° C., the deposit is drained with gentle suction in a Gooch crucible, washed until free from oil, and the residue is dissolved in hot benzene. A piece of dry cotton-wool is steeped in the hot solution, the benzene is evaporated off, the remainder of the asphalt adhering to the walls of the vessel is wiped off with cotton-wool, and the whole of the cotton-wool is packed in a filtering thimble 6 to 8 cm. long and 3 to 4 cm. wide. The thimble is suspended from the cork of a flask of 300 c.c. capacity, containing 60 to 80 c.c. of absolute alcohol, and connected with a reflux condenser. The flask is immersed in a bath of boiling water to a short distance above the top of the thimble. After extraction with the alcohol for about two hours, the flask is exchanged for a similar one, containing 60 c.c. of benzene, and after boiling the latter for fifteen to thirty minutes the hot benzene solution is poured through a filter, which is then washed with benzene. The residue left on evaporation is free from oil and paraffin, and it is weighed after drying for a short time at 105° C. The percentage of asphalt found in this way is much lower than by Holde's method. Oils containing a high proportion of asphalt—say 2 to 4 or more per cent.—should be examined in this way; further, the consistence of the precipitated asphalt should be ascertained.

J. F. B.

Testing of Coal-Tar Creosote. C. E. Sage. (*J. Soc. Chem. Ind.*, 1911, **30**, 588-591.)—Coal-tar creosote should be "the product of the destructive distillation of bituminous coal, and free from admixture with other oils." The methods of sampling adopted are important. The bulk must be liquefied, and the sample (6 half-gallon quantities) removed from different parts of a tank, mixed, and filled into cans and sealed. Creosote in barrels can only be sampled with an iron, and uniformity is difficult to obtain.

Specific Gravity.—This is best observed at 60° C., 100 c.c. of the sample being weighed, and compared with water at the same temperature. *Expansion Coefficient.*—One hundred c.c. of the sample at 40° C. is warmed to 80° C. in a flask graduated from 100 c.c. to about 106 c.c. on the neck. The increase in volume should be recorded. This increase is on the average 1 per cent. for each 13·3° C. *Flash Point.*—This determination is made in the Pensky-Marten apparatus on a portion of the sample not previously heated. Oils usually flash at 186° to 190° C. *Fluidity.*—This consists in noting whether the creosote is fluid at 40° C., and by keeping 100 c.c. of the sample at 15° C. for six hours, and stirring occasionally after adding a crystal of naphthalene. The result is specified as "fluid," "quite solid," or "some salts deposited," etc., as the case may be. *Solubility in Benzene.*—Insoluble carbonaceous matter should not exceed 0·25 per cent. This is determined by filtering 100 grms. of the hot creosote through a tared paper, and washing the residue with benzene, when the residue is weighed after drying. *Viscosity.*—This test is of little or no value. *Distillation.*—Fractional distillation of creosote should be carried out in a specified apparatus. The author uses a hard glass retort of 8 ounces capacity (up to the bend), the thermometer bulb being immersed in the liquid, and less than ½ inch from the bottom of the retort. The retort should be asbestos-covered, and the thermometer should have a range of 190° to 360° C. The presence of water in creosote interferes with the fractionation, so that in the case of creosotes containing more than 1 per cent. of water it should be specified that it be first distilled to remove water, any oil simultaneously distilling being separated and returned to the bulk, and the distillation of the dry oil then proceeded with. All the fractions are measured at 60° C., to insure fluidity. The measurements usually recorded are: Distillate up to 210° C., between 210° to 235°, 235° to 270°, 270° to 315°, and undistilled residue over 315° C. The water is separately measured, solvent naphtha being added, if necessary, to the mixture of water and the first fraction distilled, in order to obtain a clear reading.

Naphthalene.—The distillate below 270° C. is cooled and filtered through filter-cloths and the residue pressed between blotting-paper. *Tar Acids.*—The fractions distilling up to 315° C. are shaken out with caustic soda solution (15 per cent.), and the alkali subsequently acidified, and the freed acids measured at 60° C. The results when the test is carried out on the original creosote are inaccurate. A more precise method of stating the amount of "tar-acids" is required. *Pyridine Bases* are determined in the fractions distilling below 315° C. They are important from the point of view of the value of these materials as the basis of sheep-dips and disinfectants. *Undistilled Residue.*—This should be examined for separated "pitchy" matter, as such separation is undesirable when the creosote is to be used for impregnating timber. *Sulphonation.*—English creosote does not naturally contain any considerable proportion of paraffins, or not more than about 0·5 per cent. Blast-furnace creosote (high-boiling fractions) contains about 5 or 6 per cent. The paraffins may be detected by determination of the refractive index of the sulphonation residue.

Paraffins have $[N]_{D 20^\circ C.}$ of about 1·45 to 1·50, blast-furnace creosote distillates range from 1·48 to 1·54, and coal-tar distillates from 1·57 to 1·63, or higher.

When distilled from an 8-ounce retort (covered), with the thermometer-bulb immersed in the liquid, pure coal-tar distillates will yield fractions above 300° C., having $[N]_{D 20^\circ C.}$ above 1·60. A. R. T.

Estimation of Camphor in Smokeless Powder. Marqueyrol. (*Ann. Chim. anal. appl.*, 1911, **16**, 209-210.)—The method is based upon the fact that if a solution of camphor in ether be evaporated over sulphuric acid in a desiccator, the loss in weight determined at regular intervals (*e.g.*, ten minutes), and a curve plotted in which the periods of time form the abscissæ and the corresponding weights the ordinates, a point is reached where the curve bends abruptly. This ordinate, OM, indicates the weight of camphor in the solution. In applying the method to smokeless powder 10 grms. of the finely divided sample are extracted for about ten hours with ether in a Soxhlet apparatus, and the solution allowed to evaporate in a basin (about 65 to 70 mm. in diameter and 35 to 40 mm. in depth) which is placed in a desiccator containing sulphuric acid. The ordinate $OM = p$, corresponding to the ethereal solution, is ascertained as described, and the basin then heated at 30° to 40° C. so as to expel the camphor completely. If the weight of the residue be represented by p^1, the weight of the camphor is represented by $p - p^1$. C. A. M.

Method for the Accurate Estimation of Traces of Water in Ether. D. Tyrer. (*Proc. Chem. Soc.*, 1911, **27**, 142-143.)—It is occasionally of importance to determine with accuracy the amount of water contained in a sample of "wet" ether. A method susceptible of a great degree of accuracy can be based on the fact that the addition of a little water to ether greatly increases its solvent power for some solutes. The most suitable substance which can be used as a solute is cadmium iodide. According to Eder (*Dingl. Polytech. J.*, 1876, **221**, 189), cadmium iodide is soluble in "absolute" ether to the extent of 28 per cent. Using a very dry sample of ether, it was found by the author, however, that cadmium iodide is practically insoluble in ether. It is to be presumed that the high value obtained by Eder was due to the ether he used containing water and alcohol. In the following table is given the solubility of cadmium iodide at 12° C. in pure ether and in ether containing various quantities of water. The ether used was Kahlbaum's pure material, prepared from ethyl alcohol, and thoroughly dried by twice distilling over phosphoric oxide:

Grms. of Water in 100 of Ether.	Grms. of CdI_2 per 100 of Solvent.	Grms. of Water in 100 of Ether.	Grms. of CdI_2 per 100 of Solvent.
0·0	0·143	0·90	6·46
0·10	0·78	1·00	7·30
0·30	2·07	1·10	8·27
0·50	3·36	1·14 {satu- rated}	8·68
0·70	4·77		

The above results were read from a smoothed curve of experimental values. On inspecting the table it will be seen that an addition of 0·1 per cent. of water to dry ether increases the solubility of the cadmium iodide by about 0·64 unit. Now, as the solublitity can be determined with ease to within an error less than 0·01 unit, the percentage amount of water in the ether can be found to less than 0·001 unit. This method of analysis might be extended to the determination of traces of water in the alcohols, it being only necessary to find a suitable solute and to obtain a perfectly anhydrous alcohol.

Estimation of Formaldehyde by the Ammonia Method. A. Beythien, H. Hempel, P. Simmich, W. Schwert, and C. Wiesemann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 671-673.)—In the method of estimating formaldehyde by treating the latter with ammonia or with sodium hydroxide and ammonium chloride, and titrating the remaining alkali, the accuracy of the results obtained depends to a considerable extent on the indicator employed in the titration. Rosolic acid yields the most trustworthy results, cochineal and methyl-orange are less suitable, whilst litmus, phenolphthaleïn, and Congo-red are quite useless. W. P. S.

Estimation of Nitrogen in Liquids. D. Furnrohr. (*Wochensch. Brau.*, 1911, **28**, 257-258.)—For the estimation of nitrogen in beer worts it is often directed that a trace of yeast be added, the sugars fermented, and the liquid concentrated on the sand bath or open flame to a syrupy consistence. The addition of yeast is quite unnecessary, and of course inimical to the accuracy of the results. Evaporation over a flame is most objectionable, since it leads to caramelisation of the liquid and to erratic results, nitrogen being lost during evaporation. The author quotes analyses of the same wort, which show that evaporation of the wort at temperatures over 150° C. results in serious errors from the above cause. He has devised a constant-temperature bath which can be adjusted at 150° C., and which is capable of holding six Kjeldahl flasks, the contents of which may be left to evaporate without any danger of frothing over or loss of nitrogen by caramelisation. The mouth of each flask protrudes through the top of the bath, the rest being protected by a cylindrical screen in the body of the bath. J. F. B.

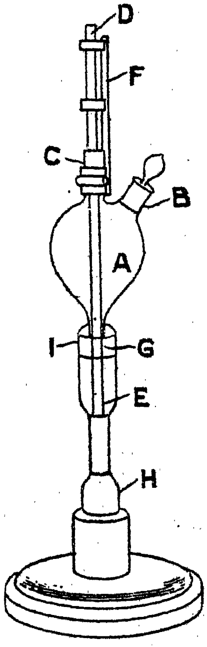
Characteristics of Different Varieties of Dammar Resin. C. Coffignier. (*Bull. Soc. Chim.*, 1911, 9, 549-561.)—The chief results obtained in the examination of different kinds of commercial dammar resin are summarised in the following table:

	Sp. Gr. at 18° C.	Melting-Point.	Acid Value.	Saponification Value.	Insoluble in—					
					Alcohol.	Ether.	Benzene.	Acetone.	Carbon Tetrachloride.	Amyl Acetate.
		°C.			Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Padang dammar (Sumatra)	1·036	95° (soft at 55°)	31·4	33·7	20·30	4·50	soluble	14·70	soluble	6·20
Borneo dammar ...	1·048	120° (soft at 70°)	35·1	64·5	23·60	9·60	7·50	20·40	8·40	10·10
Singapore dammar	1·057	95° (soft at 55°)	30·1	39·3	19·10	1·00	soluble	14·00	2·50	4·10
Pontianac dammar	1·025	110° (soft at 65°)	19·9	30·9	22·40	4·10	soluble	16·40	soluble	4·60
Sumatra dammar...	1·004	190° (soft at 115°)	59·6	64·5	45·50	37·90	18·30	45·30	31·60	30·00
Batjan dammar ...	1·032	105° (soft at 60°)	18·5	19·6	32·80	3·20	3·10	21·30	11·40	7·30

C. A. M.

Action of Permanganate on Organic Substances. J. Hetper. (*Zeitsch. anal. Chem.*, 1911, 50, 343-370.)—The author has studied the action of permanganate on nearly one hundred commonly occurring organic substances under the following conditions: Into a 200 c.c. flask 25 c.c. of a $\frac{N}{5}$ or $\frac{N}{2}$ permanganate solution (containing 20 to 40 grms. of phosphoric acid per litre) is introduced, and to this is added a measured quantity of a solution of the substance to be tested, preferably such a quantity as will reduce not much more than 10 c.c. of the permanganate. The volume of the mixture is made up to about 100 c.c. with water, and the flask placed on a vigorously boiling water-bath in a ring which is nearly as large as the maximum diameter of the flask. With volatile substances a reflux condenser is necessary. The heating is continued for one and a half to two hours, the contents of the flask being shaken every half-hour or so. At the end of this time the colour of the permanganate is discharged by addition of 25 c.c. of $\frac{N}{5}$ or $\frac{N}{2}$ oxalic acid solution (containing 50 grms. sulphuric acid per litre), and the excess of oxalic acid is then titrated with permanganate. Treated in this way a considerable number of important organic compounds decompose almost exactly the amount of permanganate which corresponds to oxidation to CO_2, H_2O (and NH_3). Ethyl alcohol is oxidised quantitatively to acetic acid, which is not further oxidised, and in general the methyl group when directly attached to another carbon atom resists oxidation, with the result that there is another large class of compounds which absorb almost exactly 4 atoms less oxygen per molecule than corresponds to complete oxidation to carbon dioxide and water. Among such compounds are lactic acid, toluidine, rosolic acid, and antipyrin. The resistance of the methyl group to oxidation is also shown by the behaviour of methyl alcohol, the oxidation of which is far from complete at the end of two hours, although formaldehyde and formic acid are rapidly oxidised to carbon dioxide and water. The author expressly disclaims any attempt to overestimate the importance of his work, but he points out that the analyst sometimes finds himself in possession of an aqueous solution which contains but one organic substance which reduces permanganate, and his tables show with what degree of exactness many such substances can be estimated. They also afford a means of establishing the purity or identity of some substances by the use of a very small quantity of material. G. C. J.

Proposed Uniform Method of carrying out comparative Tests for the Valuation of Raw Rubber. F. Frank. (*Gummi-Zeit.*, 1911, **25**, 990-992; through *J. Soc. Chem. Ind.*, 1911, **30**, 560.)—The proposals described were made to the German section of the International Rubber Testing Committee, and comprise viscosity determinations of solutions of the raw rubber, and vulcanisation tests. The apparatus described for the determination of viscosity consists of a pear-shaped glass vessel, A, with side tubulure, B (fitted with a ground-in stopper), and provided with an aluminium valve-rod, D, sliding through guides, and ground to fit the lower opening of the glass vessel, which ends in a metal tube, E, forming the outlet. The pear-shaped solution-holder, A, fits, by means of a ground-glass joint, G, into the top of the graduated receiver, H. The escape of air from this vessel while the solution is flowing into it is provided for by means of holes in the ground-glass joint at I. In carrying out the test, 200 to 500 grms. of the raw rubber are cut up, softened in hot water for thirty minutes, washed as rapidly and with as little working as possible on the rollers, run into a sheet "as thin as paper," and dried in a dark room without artificial heat. The washing process should not occupy more than twenty minutes. Any variations in details from the standard method must be carefully noted. The solution recommended is a 3 per cent. solution in commercial xylol of sp. gr. 0·867 to 0·869 at 15° C., 90 per cent. distilling between 135° and 140° C. The sample—9 to 15 grms. of dry rubber—is cut up fine, allowed to soak in about 100 c.c. of xylol for a short time, then stirred, and the final weight made up by the addition of more xylol, after which the solution



is agitated at intervals until homogeneous. This should not take more than thirty hours in all. For the preparation of the solution a dark glass jar of about 1 litre capacity, fitted with a metal screw-cap through which a stirrer passes, or simply a brown glass litre bottle, with glass stopper, may be used. The solution vessel should in either case be tared before use. Measurements are carried out at 20° C. The pear-shaped vessel is filled to the 200 c.c. mark, the valve-rod lifted, and the time taken for the solution to fill the receiver up to the 100 c.c. mark (in a footnote 200 c.c. is recommended) is accurately noted. A second determination should be carried out after the solution has stood in a dark place for eight to ten days. It is suggested that glycerol of sp. gr. 1·25 should be used to standardise the apparatus. For the vulcanisation tests the remainder of the rubber sample is mixed with eight or ten per cent. of sulphur and vulcanised in the form of a sheet 5 mm. thick, either always in the press or always in a vulcanising pan, for either one hour at 4 atmospheres, or one hour at 3 atmospheres. The vulcanised sheet is tested chemically for combined sulphur—difference between total and acetone-soluble sulphur—and mechanically, in the form of rings of 5 by 4 mm. section, punched or cut out from the sheet. The Schopper-Dalén apparatus is recommended for the mechanical tests by the members of the German section, but it is not desired to enforce the use of this apparatus.

Ceará Rubber. (*Bull. Imp. Institute*, 1911, **9**, 1-8.)—Specimens of Ceará rubber from various sources gave the following results on analysis:

	RAW MOIST RUBBER FROM—								
	CEYLON.	UGANDA.		EAST AFRICA PROTECTORATE.		NYASALAND.	THE SUDAN.	SOUTHERN NIGERIA.	
		1.	2.	1.	2.	(Dry Washed Rubber.)		1.	2. (Dry Washed Rubber.)
	%	%	%	%	%	%	%	%	%
Moisture	0·9	4·8	3·5	12·4	10·0	—	2·2	6·4	—
Caoutchouc ...	91·3	72·8	86·1	58·9	59·8	78·6	80·1	62·8	84·1
Resin	3·1	7·6	5·7	10·5	8·7	10·8	5·8	3·4	7·8
Proteins	3·4	11·9	3·6	12·1	13·9	8·4	9·8	22·4	6·5
Insoluble matter	—	—	—	6·1	7·6	—	—	5·0	—
Ash	1·3	2·9	1·1	2·4	4·0	2·2	2·1	2·6	1·6
Loss on washing ...	—	—	—	—	—	4·9	—	—	21·7
Physical properties	Very satisfactory	Very satisfactory	Very satisfactory	Fair	Poor	Fair	Good	Unsatisfactory	Fair

A. R. T.

Effect of the Glycerol Method of Saponification upon the Fatty Acids. H. Mielck. (*Chem. Zeit.*, 1911, **35**, 668.)—A comparison of the values of the fatty acids obtained by Zoul's method of saponifying fats with the aid of glycerol (*Chem. Zeit.*, 1911, **35**, *Rep.*, 23) and by the ordinary method of saponification with alcoholic alkali, has shown that the former causes slight oxidation. Thus the iodine values (Wijs) of the fatty acids from bone fat ranged from 49·6 to 51·4 in the case of the glycerol method as against 54·7 to 54·9 in the case of the ordinary method. There were also marked differences in the solidification points. If a carbon tetrachloride solution of the fatty acids obtained by the glycerol method is allowed to stand for twelve hours, it yields a dark brown deposit. Hence in the author's opinion the method of saponification with alcoholic alkali ought to be used when a "titre" estimation of the fatty acids is required. C. A. M.

INORGANIC ANALYSIS.

Analysis of Aluminium and Aluminium Alloys. G. Kleist. (*Chem. Zeit.*, 1911, **35**, 668-669.)—Silicon is estimated as in Thorn-Smith's method (*Chem. Zeit.*, 1911, **35**, *Rep.* 188), but without the addition of sulphuric acid. For the estimation of iron from 2 to 5 grms. of the turnings are boiled with about 100 c.c. of dilute sodium hydroxide solution, the resulting solution rendered strongly acid with sulphuric acid (1 : 1) and filtered, and the filtrate diluted to about 600 c.c. and titrated with standard permanganate solution. Copper and tin are estimated together by precipitation with hydrogen sulphide in the filtrate from the silicon, and ignition of the precipitate in a porcelain crucible. If tin is present it is estimated as described below. The filtrate from the copper and tin is concentrated to about 100 c.c., and the iron precipitated by means of sodium hydroxide solution. In the presence of much iron (over 1 per cent.) the precipitate must be dissolved and the precipitation repeated. Traces of zinc are precipitated from the alkaline filtrate by means of hydrogen sulphide.

Aluminium alloys usually contain up to about 10 per cent. of copper, 6 per cent. of tin, and up to 10 per cent. (rarely up to 30 per cent.) of zinc, together with silicon and iron as impurities. The copper, zinc, silicon, and iron are estimated as in the case of pure aluminium. For the estimation of the tin from 1 to 2 grms. of turnings are treated with about 150 c.c. of hydrochloric acid (sp. gr. 1·19), and the flask immediately closed with a perforated rubber stopper, and heated until solution of the metal is complete. The boiling solution is then titrated with iron chloride solution, which has been standardised upon pure tin. The amount of tin thus found is calculated into tin oxide, and deducted from the weight of the combined copper and tin oxides previously estimated. Or the combined copper and tin precipitate may be treated with 2 c.c. of nitric acid (1 : 4), the liquid filtered from the tin oxide, and the filtrate treated with about 5 c.c. of sulphuric acid (1 : 1), diluted to about 120 c.c., and electrolysed at 60° to 70° C. The filtrate from the copper and tin is concentrated to about 100 c.c., rendered alkaline with ammonia, and treated with dilute sulphuric acid (1 : 1) until all aluminium hydroxide has dissolved, care being taken to avoid an excess of acid. The solution is diluted to about 400 to 500 c.c., and the zinc precipitated by means of a current of hydrogen sulphide. The precipitate is washed with water acidified with sulphuric acid and containing some hydrogen sulphide and ammonium sulphate, and then dissolved in hot dilute hydrochloric acid. The solution is rendered faintly alkaline with ammonia, boiled and filtered, and the filtrate slightly acidified with sulphuric acid and again treated as described.

C. A. M.

Separation of Aluminium from Beryllium. Wunder and N. Chéladzé. (*Ann. Chim. anal. appl.*, 1911, **16**, 205-209.)—Two methods, based upon the solubility of beryllium hydroxide in a concentrated solution of ammonium carbonate, are shown to give incorrect results. The following method is satisfactory, and may be used in the presence of iron, which is separated simultaneously with the beryllium. The mixed hydroxides are dissolved in hydrochloric acid, the solution evaporated to dryness in a platinum basin on the water-bath, and the residue taken up in the smallest possible quantity of water. A saturated solution of sodium or potassium hydroxide is now added drop by drop, with continual stirring, until the gelatinous mass first obtained is just redissolved. A large excess (400 c.c.) of water is then added, and the liquid boiled for an hour, the evaporated water being replaced from time to time. The precipitated beryllium hydroxide is collected, washed with boiling water, ignited, and weighed as beryllium oxide (BeO). The filtrate is acidified, and the aluminium precipitated with ammonia. The results in the test estimations quoted agree closely with the theoretical amounts. It is essential, however, to have sufficient sodium or potassium hydroxide to keep the whole of the aluminium in solution.

C. A. M.

Separation and Estimation of Barium in Presence of Calcium and Magnesium by the Action of Acetyl Chloride and Acetone on the Mixed Chlorides. F. A. Gooch and C. N. Boynton. (*Zeitsch. anorg. Chem.*, 1911, **70**, 400-407.)—The aqueous solution, containing about 0·1 grm. of barium chloride, is first concentrated to 3 c.c. or less; to this 30 c.c. of a mixture of acetyl chloride and acetone (1 : 4) is added at the rate of 2 drops a second, the mixture being kept cool in running water and well stirred. The separated barium chloride is filtered off on asbestos in a Gooch crucible, washed with acetone, and dried at 135° C., or ignited at a low red heat. Barium may be separated in this way from ten times its weight of calcium or magnesium, and estimated with an error not exceeding ±0·1 per cent. It is important that the mixture be kept cool during the addition of the acetyl chloride to the aqueous solution, and, consequently, the rate of this addition should not exceed that stated.

Strontium cannot be quantitatively separated from barium nor from calcium and magnesium by similar means.

G. C. J.

Phenomenon of Occlusion in Precipitates of Barium Sulphate, and its Relation to the Exact Determination of Sulphate. J. Johnston and L. H. Adams. (*J. Amer. Chem. Soc.*, 1911, **33**, 829-845.)—The investigation of Allen and Johnston (ANALYST, 1910, **35**, 331) has been continued, and on the knowledge gained attempts have been made to find a direct method for the determination of sulphate, which should be generally applicable, exact, and require only small and easily determined corrections. The belief is expressed that there is little chance of discovering any exact method which shall be generally applicable and require no corrections, and yet not owe its accuracy to a compensation of errors. Where, however, the composition of the solution is approximately known—a condition which frequently obtains in practice—the following calibration method will yield results accurate to 0·05 per cent.: To the solution (300 c.c. for a precipitate to weigh 2 grms.) 50 c.c. of concentrated hydrochloric acid is added, and the mixture heated to boiling. A 10 per cent. solution of barium chloride is then added at such a rate that about four minutes is required to run in the necessary 22 c.c. The mixture is evaporated to dryness on the water-bath, the residue taken up with hot water, and the barium sulphate filtered off, washed free from chloride, ignited carefully to avoid reduction, and heated to constant weight over a bunsen-burner. The necessary correction is determined by a concurrent calibration of the method—that is, by dissolving an equivalent weighed amount of pure sodium or potassium sulphate (dried for an hour at 200° C.) in a medium, such that the resulting solution is as nearly as may be of the same composition as the solution to be analysed, and determining the sulphate in this solution as already described. The difference between the calculated amount of barium sulphate and that actually found is the correction to be applied to the weight of the precipitate obtained in the actual analysis. The correction is usually small, and represents the balance of errors which are individually small under the conditions described. The use of a large quantity of acid reduces the occlusion of soluble sulphates by the precipitate, and gives to this a coarsely granular form, which facilitates all the subsequent operations, whilst very little barium sulphate passes into the filtrate, as the acid is almost wholly eliminated during the evaporation. G. C. J.

New Reaction of Bromine. G. Denigès. (*Bull. Soc. Chem.*, 1911, 9, 542-544.)—A delicate test for bromine is based upon the fact that it gives a purple coloration with the derivatives obtained by reducing strychnine with hydrogen. A suitable reagent may be prepared by mixing together 5 c.c. of a 1 per cent. aqueous solution of strychnine, 5 c.c. of hydrochloric acid (sp. gr. 1·18 to 1·19), and from 4 to 5 grms. of pure granulated zinc, heating the mixture to the boiling-point, then leaving it for five to ten minutes, at the ordinary temperature, cooling it in water and decanting the supernatant liquid. When 5 c.c. of an aqueous solution of bromine, containing not more than 1 grm. per litre, are shaken with 1 c.c. of this reagent, a purple coloration is soon developed, and the spectrum of the liquid shows a characteristic absorption band in the yellow. The test is capable of detecting 0·01 mgrm. per c.c. of solution, but in the case of such dilute solutions it is advantageous before adding the reagent to add to the liquid one drop of a 2 to 4 per cent. solution of potassium hydroxide, and to compare the tint with that given by a blank solution containing the same quantity of alkali and reagent in 5 c.c. of water. The reaction is quite distinct from that given by chlorine and other oxidising substances. Bromine may be detected in the atmosphere by passing a slow current of air through 5 c.c. of $\frac{N}{10}$

sodium or potassium hydroxide solution, and then adding 1 c.c. of the reagent. The analogous derivatives of brucine give an olive coloration, changing to yellow both with bromine and with chlorine. C. A. M.

Microscopical Examination and Identification of Carbon. G. A. Roush. (*J. Ind. Eng. Chem.*, 1911, **3**, 368-372.)—The author has applied the principles of metallography in the examination of carbon such as is used in the electrical industry. Since the orientation of the particles of carbon varies with the shape of the particles and with the shape of the die through which they are forced during manufacture into electrical carbons, at least three sections should be prepared from the block under test, and preferably these three sections should be perpendicular to the three principal axes of the block. The samples, after being removed from the block by means of a hack-saw, are first flattened by touching against an emery wheel or dressed with a file. The flat surface is then polished with No. 1 or No. 0 emery paper and this is followed by successive grades of Crocus paper from No. 0 to No. 0000. The polished surface is then either "heat-etched" or "relief-polished." Heat-etching is accomplished by raising to a red heat in the flame of a blast lamp, allowing the softer particles to oxidise in the open air until the gloss of the polished surface just disappears, and then cooling rapidly with the bellows. Relief-polishing is effected with chamois leather or cloth. It serves the same purpose as heat-etching in wearing away the softer constituents of the block and leaving the harder ones in relief. The samples are next mounted on microscope slides by means of sealing-wax, and examined by reflected light, under a magnification of 70 and of 200 diameters. Occasionally a higher magnification is useful, when a sample is found in which the raw materials have been ground very finely. The paper is accompanied by thirty microphotographs which show that petroleum coke, retort carbon, and natural graphite, can as a rule be readily distinguished, and that the proportions in which they occur in a mixture can be approximately determined. G. C. J.

Detection of Chromium in Steel. V. Stanck. (*Chem. Zeit.*, 1911, **35**, 308.)—The metal is dissolved in dilute sulphuric acid (1 : 5), the solution is boiled after the addition of ammonium persulphate, and the presence of chromium is then detected by shaking the solution with hydrogen peroxide and ether.

Estimation of Chromium in Chrome Ironstone. O. Nydegger. (*Zeitsch. angew. Chem.*, 1911, **24**, 1163-1164.)—The ore is opened up by means of a borax flux, which is prepared in advance in quantity to avoid risk of mechanical loss by evolution of carbon dioxide during the fusion. The flux is prepared by fusing together 2 parts of anhydrous sodium carbonate and 1 part of borax glass, and continuing to heat with the blowpipe flame until a tranquil fusion results. The cooled mass is coarsely powdered, and, since it is extremely hygroscopic, stored in air-tight vessels. Of this flux, 5 grms. is mixed with 0·5 grm. of the ore in a platinum crucible, which is lightly covered and heated in a powerful Bunsen flame until, on tilting the crucible, no particles can be seen at the bottom. A rotatory motion imparted to the crucible at frequent intervals hastens the process, which is usually complete in one to two hours. The cooled crucible is with its contents digested with 300 to 400 c.c. water containing 4 c.c. of sulphuric acid, and the crucible, when clean, removed from the beaker. A trace of chromic oxide, always present in the product of the fusion, is completely oxidised by boiling with 0·2 grm. of potassium persulphate, which also precipitates any manganese as peroxide. The boiling is continued for thirty to forty minutes to destroy the excess of persulphate, and the solution filtered from manganese peroxide. The chromic acid in the filtrate is then estimated by titration with a solution of ferrous ammonium sulphate standardised on pure potassium bichromate. The sulphuric acid solution of 5 grms. of the flux usually has a measurable oxidising action on a ferrous solution, and this should be determined once for all on each batch of flux prepared. G. C. J.

Analysis of Calcium Cyanamide. A. Monnier. (*Chem. Zeit.*, 1911, **35**, 601-602.)—Methods are given for the estimation of the cyanamide nitrogen and the total nitrogen in calcium cyanamide. In the case of the cyanamide nitrogen, the author finds that Caro's method (precipitation with silver nitrate in ammoniacal solution) yields low results, probably owing to the slight solubility of silver cyanamide in ammonia. He therefore proposes that 25 c.c. of a 1 per cent. solution of the calcium cyanamide be mixed with 10 c.c. of formic acid and evaporated almost to dryness; the residue is then dissolved in water, diluted to a volume of 25 c.c., and 10 c.c. of the solution are treated in a nitrometer with sodium hypobromite solution. The treatment with the formic acid results in the formation of urea according to the equation: $CN.NCa + H_2O + 2HCOOH = CO(NH_2)_2 + Ca(COOH)_2$, and the nitrogen liberated from the urea is a measure of the cyanamide nitrogen. As regards the estimation of the total nitrogen, it is stated that the ordinary Kjeldahl method yields 1 per cent. less nitrogen than is found when the digestion is carried out after the addition of salicylic acid and zinc dust, and the author concludes that this 1 per cent. of nitrogen is present in the form of a compound which has not yet been identified; this compound is converted by oxidising agents, such as hydrogen peroxide, into nitrites and nitrates.

A. Stutzer (*Chem. Zeit.*, 1911, **35**, 694), in commenting on the above paper, states that Caro's method yields correct results if the nitrogen be estimated in the silver precipitate; owing to the formation of double and basic compounds, the titration of the silver, as carried out by Monnier, does not accurately measure the amount of cyanamide nitrogen. He also states that he has always found the results obtained by the ordinary Kjeldahl method to agree with those yielded by the salicylic acid-zinc dust method. W. P. S.

Separation of Cerium by Potassium Permanganate. E. J. Roberts. (*Chem. News*, 1911, **103**, 303-304.)—One of the most useful methods for the separation of cerium from other rare-earth metals depends upon its oxidation by permanganate—

$$3Ce_2O_3 + 2KMnO_4 + H_2O = 6CeO_2 + 2KOH + 2MnO_2$$

—the reaction taking place in a cerous nitrate solution when sufficient alkali is present to combine with the nitric acid set free. The author has found the following to be the best mode of operating to insure efficient separation: The solution used for precipitation should contain the reagents in the proportion of 1 molecule of permanganate to 4 molecules of dry sodium carbonate in any convenient quantity of water. The solution of rare-earth nitrates, free from other acid radicles, is boiled and neutralised with sodium carbonate solution. Potassium permanganate solution is then added in small quantities till the red colour is just permanent, when the mixed permanganate and alkali solution is run in very slowly with constant stirring, the solution being kept near the boiling-point throughout the process. A faint permanganate colour is maintained, a little permanganate solution being run in, if necessary, from time to time. When the precipitation of the cerium is nearly complete, the colour of the solution is bleached more slowly after each addition of the precipitant, and the acidity of the liquid should be tested occasionally with litmus-paper, small quantities of alkali or mixed solution being added till the liquid is nearly neutral and still faintly coloured. The liquid is heated and stirred for ten minutes and filtered hot, and the precipitate washed with boiling water till the washings give no precipitate with ammonia. To insure complete removal of the cerium, the liquid must finally be made quite neutral, the filtrate then giving no test for this metal with hydrogen peroxide.

The test is made after boiling the filtrate with alcohol to remove permanganate. The precipitate of cerium and manganese hydroxides is dissolved in hydrochloric acid, the solution diluted, precipitated with oxalic acid, and the cerium oxalate reconverted to nitrate. On repeating the above process with this material, a pure cerium salt is obtained. The neodymium and praseodymium present in the cerium precipitate obtained after the first separation cannot be wholly removed by a nitric acid treatment of the precipitate. A. R. T.

Estimation of Manganese by the Bismuthate Method. W. F. Hillebrand and W. Blum. (*J. Ind. Eng. Chem.*, 1911, **3**, 374-376.)—It is pointed out that the theoretical factor for the ratio $Na_2C_2O_4 : 2Mn$ is 0·16397, not 0·16024, as stated by Brinton (ANALYST, 1911, 305). Brinton described experiments which led him to recommend the use of the empirical factor 0·1656 in calculating the manganese titer of permanganate. Brinton's reasoning is not vitiated by the above arithmetical error, and his results, so far as they go, do support the use of the factor recommended by him. But the authors describe experiments which support equally well the use of the theoretical factor, and they point out that Brinton's chain of reasoning assumes the accurate standardisation of his manganese sulphate solution, and an error of less than 1 per cent. in the figures supplied with samples by the Bureau of Standards, with which they are associated. Brinton's method of standardising his manganese sulphate solution would include as manganese any iron, calcium, or magnesium, and the authors disclaim a higher accuracy than 1 per cent. for the figures supplied by the Bureau of Standards, so far as these figures relate to iron with less than 1 per cent. of manganese. It is doubtful whether the manganese content of such material can yet be ascertained with a greater accuracy than 1 per cent., which is exactly the amount by which Brinton's factor differs from the true theoretical factor.

G. C. J.

Use of Ether in the Analysis of Metals. F. Mylius and C. Hüttner. (*Ber.*, 1911, **44**, 1315-1327.)—Rothe (*Mitt. d. Kgl. Techn. Versuchsaust.*, 1892, 132) has shown the use that can be made of ether in the analysis of steel; and more recently Mylius (Analyst, 1911, 303) has worked out a very exact method for the assay of gold bullion, in which ether is used to separate the greater part of the gold from the other metals. The present paper describes the behaviour of the chlorides of thirteen other metals when these are shaken with equal volumes of ether and of water or hydrochloric acid of varying strength. The figures in the table show the percentage of the metal which passes into the ethereal layer:

Metallic Chloride.	Water.	Hydrochloric Acid, Percentage of HCl—								
		0·18	1·0	2·5	5·0	7·5	10·0	12·5	15·0	20·0
$FeCl_3$	—	—	Trace	—	0·1	—	8·00	—	92·0	99·00
$AuCl_3$	—	40·3	84·0	—	98·0	—	98·00	—	—	95·00
$AsCl_3$	Nil	—	0·2	—	0·7	—	7·00	—	37·0	68·00
$SbCl_5$	Nil	—	Trace	—	2·5	—	6·00	—	22·0	81·00
$TeCl_4$	Nil	—	Trace	—	0·2	—	3·00	—	12·0	34·00
$SnCl_4$	Nil	—	0·8	—	10·0	—	23·00	—	28·0	17·00
$SbCl_3$	Nil	—	0·3	0·7	8·0	23·0	22·00	16·0	13·0	6·00
$HgCl_2$	69·4	—	13·0	—	—	—	0·40	—	—	0·20
$CuCl_2$	Nil	—	Trace	—	—	—	0·05	—	—	0·05
$ZnCl_2$	Nil	—	Trace	—	—	—	0·03	—	—	0·20
$NiCl_2$	—	—	Trace	—	—	—	Trace	—	—	Trace
$PtCl_4$	Trace	—	Trace	—	—	—	Trace	—	—	Trace
$IrCl_4$	Trace	—	Trace	—	—	—	0·02	—	—	5·00
$PdCl_2$	0·02	—	Trace	—	—	—	Trace	—	—	Nil
$PbCl_2$	Nil	—	Nil	—	—	—	Nil	—	—	Nil
$AgCl$	Nil	—	Nil	—	—	—	Nil	—	—	Nil

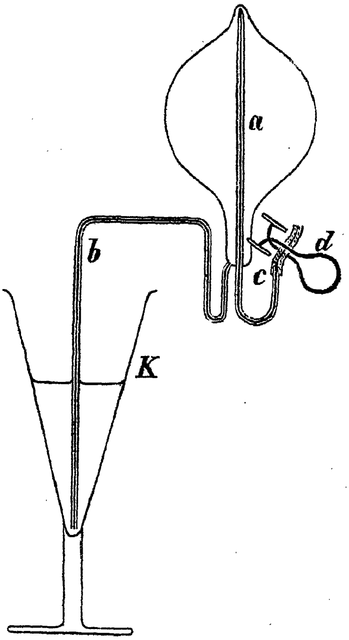
The solubility of arsenious chloride and antimonic chloride in ether is not sufficiently high to give the ether process any advantage over other methods for separating these metals. The table is, however, of value to the analyst using Rothe's method for iron, or that of Mylius for gold, as it shows what other metals may be expected to pass into the ether with the iron or gold. Since over 90 per cent. of any gold present passes into the ethereal layer, whether the hydrochloric concentration be 2 per cent. or 20 per cent., this concentration may in practice be so chosen as to keep back any particular impurity. In general it should be kept low, as the solubility in ether of most metallic chlorides is increased by the presence of much acid; but that of mercuric chloride is decreased, and that of antimonious chloride reaches a maximum with acid of 7·5 to 10 per cent. strength, and then falls again. Stannic chloride behaves like antimonious chloride, but the abnormality is less marked in this case.

G. C. J.

Volumetric Methods for the Estimation of Mercury depending on Reduction to Metal. F. Reinthaler. (*Chem. Zeit.*, 1911, **35**, 593-595.)—All the published methods covered by the title have been tried, and the following modifications of Feit's method (*Zeitsch. anal. Chem.*, 1889, **28**, 318) is recommended as surpassing any of them in accuracy. Like Feit's original method, it has the disadvantage that the mercury in the solution to be tested must all be in the mercuric state. To the mercuric solution, which should be nearly neutralised with caustic soda, $\frac{N}{10}$ sodium arsenite is added in quantity more than sufficient to reduce the whole of the mercury to the metallic state. A current of carbon dioxide is led into the flask by means of a tube which has a bulb blown on it, which serves to loosely stopper the flask and to keep the end of the tube just above the surface of the liquid. The flask and its contents are then heated on the water-bath for half an hour to one and a half hours, a gentle stream of carbon dioxide being continued all the time. When the reaction is complete, the precipitate should contain no large particles nor possess any green tinge, but should be a uniform dark grey "flowing" powder. The mixture is cooled, and the excess of arsenious acid titrated directly with $\frac{N}{10}$ iodine. Filtration is unnecessary, as the mercury is in globules of such a size that solution of mercury by iodine is so slow that no inaccuracy is involved by adding a distinct excess of iodine and titrating back with arsenite. This is one advantage of the method as compared with Feit's original method. The other is the increased accuracy, which is within 0·05 c.c. of the solutions used, and is attained at the expense of the small amount of trouble required to conduct the reaction in a non-oxidising atmosphere. The method is not as quick as that of Rupp (*Arch. Pharm.*, 1905, **243**, 300), and Rupp's method has the advantage that it is applicable to mixtures of mercuric and mercurous salts, which are first reduced to metal by some reducing agent without action on iodine in acid solution, the mercury being then dissolved in excess of $\frac{N}{10}$ iodine solution and the excess of iodine determined by means of thiosulphate. But, as originally described, Rupp's method does not yield results of the highest accuracy, and the modifications of it (for instance, that of Muller, *Bull. Soc. Chim.*, 1907, **1**, 1169) complicate it without notable improvement in the accuracy of the results. G. C. J.

Detection of Nitrates and Nitrites in Water. G. Denigès. (*Bull. Soc. Chim.*, 1911, **9**, 544-546.)—The hydro-strychnine reagent for bromine (see p. 427) may also be used for the detection of traces of nitrites and nitrates in water. On adding 0·5 c.c. to 10 c.c. of water containing nitrites, a rose-coloured tint is immediately developed; and if the amount of nitrous acid does not exceed 0·1 mgrm., a rapid colorimetric estimation may be made by comparison with standard samples. In the absence of nitrites, no coloration is produced by nitrates even when present in large quantity; but if the liquid be treated with half its volume of sulphuric acid (sp. gr. 1·84) free from nitrous products, a rose coloration develops on shaking. The reaction is capable of detecting 0·5 mgrm. of nitric acid per litre. In the presence of both nitrites and nitrates, the coloration will be intensified after the addition of the sulphuric acid. The nitrites may be eliminated by evaporating 10 c.c. of the water to dryness after the addition of 2 drops of ammonia solution and 3 to 4 drops of acetic acid. The residue when taken up with 10 c.c. of distilled water ought not to give any coloration with 0·5 c.c. of the reagent, but in the presence of nitrates should give a more or less intense red coloration. Chlorides do not interfere with the reaction. C. A. M.

Estimation of Nitric Oxide, and its Behaviour towards Water. L. Moser. (*Zeitsch. anal. Chem.*, 1911, **50**, 401-433.)—Nitric oxide cannot be preserved without change in contact with water; but if mercury be used as the retaining medium, the dry or moist gas can be preserved indefinitely. Nitric oxide is absorbed completely and quickly when shaken in a Hempel pipette with a saturated solution of ferrous sulphate made faintly acid with sulphuric acid; but the method fails in presence of nitrous oxide, on account of the high solubility of this gas in water, even when this is saturated with a salt such as ferrous sulphate. Previous saturation of the ferrous sulphate solution with nitrous oxide changes the large positive error to a small, but not insignificant, negative one; for the absorption of nitric oxide by the liquid reduces its capacity to hold nitrous oxide in solution, and this gas is set free, with the result that the nitric oxide may be underestimated by as much as 4 per cent. Divers' sodium sulphite method (*J. Chem. Soc.*, 1899, **75**, 82) possesses no advantage over the ferrous sulphate method, and the absorption is much slower. The gravimetric methods for the estimation of nitric oxide require such elaborate precautions to render them quantitative that they are rarely applicable. Among volumetric methods, that which makes use of permanganate has the advantage that this reagent is without action on nitrous oxide; but the apparatus recommended by Lunge (*Zeitsch. angew. Chem.*, 1890, **8**, 567), a train of bulbs, requires to have its air contents displaced by carbon dioxide, and this leads indirectly to errors which cannot be kept as low as 2 per cent. The author has devised the apparatus here illustrated, with which results are obtainable which are never more than 0·5 per cent. low. The apparatus consists of a glass bulb of 120 c.c. capacity, shaped like a glow-lamp, into which two capillary tubes are sealed, one of which, *a*, reaches nearly to the point of the bulb. The bulb is filled with the permanganate solution by charging the wineglass with 100 c.c. of $\frac{N}{10}$ permanganate and 40 c.c. 2N sulphuric acid, and applying suction at *c* by means of a water-pump. When the capillary *a* is filled with liquid up to the pinchcock, the latter is closed, and the gas burette containing the gas mixture is connected to *c* by the rubber connection. The pinchcock is opened and the gas driven over into the bulb, the pinchcock closed, and the bulb shaken whilst keeping the end of *b* sealed in the wineglass. Shaking is repeated after an interval of a few minutes, and when no further contraction takes place the permanganate is expelled from the apparatus and titrated. G. C. J.



Detection and Estimation of Nitric Acid and Nitrous Acid in Water. J. Tillmans and W. Sutthoff. (*Zeitsch. anal. Chem.*, 1911, **65**, 473-495.)—The authors give the conditions under which both nitric and nitrous acids may be detected and estimated by means of the diphenylamine reaction. Although this reaction is well known, it has been found to yield very uncertain results, principally owing to the fact that the water tested did not contain a sufficient quantity of chlorides, the presence of which is essential to the sensitiveness of the test. The concentration of the sulphuric acid employed is also of importance. Under the conditions given below, the method is stated to be capable of detecting the presence of as little as 0·1 mgrm. of nitric acid (N_2O_5) per litre; the blue coloration is fully developed in about one hour, and its intensity is exactly proportional to the amount of nitric acid present. The reagent is prepared as described previously by one of the authors (ANALYST, 1911, 67) for the detection of nitrates in milk. One c.c. of the water, to which 2 c.c. of saturated sodium chloride have been added per 100 c.c., is mixed with 4 c.c. of the reagent, the liquid being kept cool during the mixing, and after the lapse of one hour the blue coloration which develops is compared with that yielded by known quantities of nitric acid under similar conditions. If the water contains more than 2·5 mgrms. of nitric acid per litre, the blue coloration produced will be too deep for comparison, and the water must be diluted before being tested. For the detection of nitrous acid (nitrites) in water the reagent must be diluted, 200 c.c. of water being added to 500 c.c. of reagent. Five c.c. of the water are mixed with 5 c.c. of the diluted reagent, and if nitrous acid be present a blue coloration develops immediately. The presence of chlorides is not necessary, and consequently nitrous acid may thus be distinguished from nitric acid. The test is as sensitive for nitrous acid as for nitric acid, and the coloration in both cases is fairly stable. If the water under examination contains both nitrates and nitrites, the latter are estimated in a portion of the sample; the nitrites are then decomposed in another portion of the water, and the remaining nitrate is estimated. For the removal of the nitrites, 100 c.c. of the water may be mixed with 5 drops of dilute sulphuric acid and 0·2 grm. of urea, and allowed to stand for about sixteen hours; the nitrous acid is thus decomposed according to the equation:—

$$2HNO_2 + CO(NH_2)_2 = CO_2 + 2N_2 + 3H_2O$$

whilst the nitric acid is unaffected. For the same purpose 100 c.c. of the water may be evaporated to a volume of 25 c.c. after the addition of ammonium chloride; in this case the nitrous acid is decomposed according to the equation:—

$$NaNO_2 + NH_4Cl = NaCl + N_2 + 2H_2O.$$

W. P. S.

Estimation of Phosphoric Acid as Magnesium-Ammonium Phosphate. G. Jörgensen. (*Zeitsch. anal. Chem.*, 1911, **50**, 337-343).—The author defends his method (ANALYST, 1909, **34**, 392) against the criticisms of Bube (*Zeitsch. anal. Chem.*, 1910, **49**, 525).

G. C. J.

Application of the "Glow Reaction" to the Detection of the Platinum Metals. L. J. Curtman and P. Rothberg. (*J. Amer. Chem. Soc.*, 1911, **33**, 718-724.)—Platinum metals, especially when in a finely divided state as obtained by reduction of their salts, possess the property of condensing relatively large volumes of gases upon their surfaces at ordinary temperatures, and the reaction is frequently so energetic that the metallic "sponge" glows intensely. The authors have applied this "glow reaction" to the detection of these metals when a mixture of air and illuminating gas is passed over the finely divided metal.

The substance to be tested is brought into solution by the usual methods, and about 0·2 c.c. absorbed in a piece of thin asbestos-paper by alternately dipping the "paper" in the solution and heating till the required volume has been absorbed. The moist paper, held in tongs, is heated to redness in the Bunsen flame, removed from the source of heat, and while hot, but after the redness has ceased, it is brought into the stream of a mixture of illuminating gas and air from a Bunsen burner. If platinum is present, the asbestos will begin to glow. This glow must last for some time, and may be renewed, after dying out, by reheating the asbestos and again holding it in the gas. The asbestos-paper must be very thin (0·5 cm. × 3·0 cm. and 0·3 mm. thickness) and the burner arranged to supply a good mixture of gas and air, without too great pressure. If desired, the intensity of the glow may be increased by passing the gases through a warmed burner.

The test is sensitive to extremely small quantities of the metals, the limits found being: Platinum, 0·002 mgrm.; palladium, 0·0005 mgrm.; rhodium, 0·0009 mgrm.; and iridium, 0·005 mgrm. The presence of other substances does not, for practical purposes, interfere with the test, which may therefore be applied directly to a solution without preliminary treatment for the separation of other substances. Compounds which, however, leave a heavy residue on ignition (alkalis, etc.), impair the test to the extent that quantities of platinum near the limit will not glow in their presence.

The solution to be tested must, of course, be concentrated so that 0·2 c.c. will contain sufficient metal to give the test, and failure to obtain a glow-reaction shows that the metal is not present in the volume absorbed in an amount equal to that indicated as the limit for that metal. Osmium and ruthenium salts do not respond to the test. As a test for platinum the method is rapid and sensitive. A. R. T.

Sodium Thiosulphate as Standard in Alkalimetry. W. Feld. (*Zeitsch. angew. Chem.*, 1911, **29**, 1161-1163.)—When excess of mercuric chloride is added to a solution of thiosulphate, the latter is oxidised in accordance with the following equation: $Na_2S_2O_3 + 2HgCl_2 + H_2O = Hg_2Cl_2 + Na_2SO_4 + 2HCl + S$. It is suggested, therefore, that sodium thiosulphate, which is obtainable commercially in a state of great purity, affords one of the best available standards for controlling solutions intended for use in alkalimetry. In standardising $\frac{N}{10}$ alkali, about 0·5 grm. of crystallised thiosulphate ($Na_2S_2O_3.5H_2O$) is accurately weighed, dissolved in water, and the solution added to about 40 c.c. of a cold saturated solution of mercuric chloride. Excess of mercuric chloride is essential, as otherwise black mercuric sulphide separates. Five minutes is allowed for the reaction to complete itself, and then about 30 c.c. of 4N ammonium chloride is added to prevent the precipitation of yellow mercuric oxide by the caustic alkali which would mask the colour of the indicator. Methyl orange is added, and the titration with the caustic alkali proceeded with. The error of the method is ± 0·05 c.c. of $\frac{N}{10}$ alkali. G. C. J.

Estimation of Sulphides in Alkali-Metal Cyanides. E. C. Rossiter. (*J. Soc. Chem. Ind.*, 1911, **30**, 583-588.)—The author has experimentally examined three methods for the determination of sulphides in sodium and potassium cyanides —namely, the Ewan method (ANALYST, 1909, **34**, 117), the method of Williams (*J. Soc. Chem. Ind.*, 1906, **26**, 137), and the "mercury method," in which the sulphide is separated and weighed as mercuric sulphide, obtained by adding mercuric chloride to a solution of the sample, and warming till coagulation of the precipitate takes place. The last-named method is not accurate to more than about 0·005 per cent. As a result the author has devised a modified lead nitrate method, whereby the sulphide in cyanides can be determined to about 0·001 per cent., or one part in 100,000. The cyanide is first tested by the above-mentioned Ewan process, and from the result obtained the quantity of lead nitrate required to precipitate the sulphide in 100 grms. of the sample is calculated. This quantity of finely powdered lead nitrate (dried over sulphuric acid), plus 10 per cent. excess, is added to 100 grms. of the sample and 200 c.c. of air-free water. The mixture is stirred and slightly warmed till all cyanide is dissolved; a drop of standard sodium sulphide is then added to a little of the solution, withdrawn and first filtered, to determine the presence of an excess of lead. This excess of lead is then estimated by adding standard sodium sulphide solution till a small quantity of the solution after filtration shows no trace of lead on adding a drop of sulphide solution. If only 0·015 per cent. of sulphide is present in the sample, 150 or 200 grms. are taken for the determination. A. R. T.

Detection and Estimation of Thorium by Means of Iodic Acid. (Separation of Thorium from Scandium.) R. J. Meyer. (*Zeitsch. anorg. Chem.*, 1911, **71**, 65-69.)—The use of iodic acid in the quantitative separation of thorium from cerium has been described by Meyer and Speter (ANALYST, 1910, **35**, 281). In making use of the same reagent for the detection of traces of thorium in presence of much cerium the conditions of success are sufficient concentration of nitric acid (to retain the cerium in solution) and of iodate to decrease the solubility of the thorium iodate in the acid solution. To 2 c.c. of the solution to be tested, which must contain no free hydrochloric acid, there is added 5 c.c. of a solution (I.) made by dissolving 10 grms. potassium iodate in 100 c.c. dilute nitric acid (1 : 2). Under these conditions the precipitation of thorium is almost complete, but the precipitate may contain iodates of cerium and other earth metals, according to their concentration in the original solution. To the mixture in the test-tube, therefore, there is next added 10 c.c. of a weaker iodate solution (II.) made by dissolving 4 grms. potassium iodate in 500 c.c. dilute nitric acid (1 : 9). The mixture is heated to boiling, when the iodates of cerium, etc., dissolve, whilst that of thorium remains undissolved if the original 2 c.c. of solution taken for the test contained as much as 0·25 mgrm. of thorium. Zirconium is precipitated by iodic acid, but the iodate of zirconium can be distinguished from that of thorium by its solubility in oxalic acid. The precautions requisite in quantitative work, to overcome the tendency of cerous salts in nitric acid solution to become oxidised to the ceric state, are unnecessary, as such oxidation proceeds so slowly that its extent is negligible during the time occupied by a qualitative test. But since ceric iodate is difficultly soluble in nitric acid, the solution to be tested should first be boiled with sulphurous acid to reduce any ceric salts.

Separation of Thorium from Scandium.—Scandium iodate being soluble in nitric acid, the iodate method affords a means of separating scandium from thorium. The total earths from 3 to 4 grms. of mineral are separated by the oxalic acid method and weighed as oxides. The oxides are dissolved in hydrochloric acid, the solution evaporated to dryness, the residue taken up with water, and thorium and scandium precipitated in boiling solution by means of ammonium thiosulphate. The precipitate is redissolved in hydrochloric acid and reprecipitated with thiosulphate to eliminate completely the other cerium and yttrium earths. The thiosulphate precipitate is then decomposed by means of nitric acid, and the solution is filtered from the separated sulphur and evaporated to dryness. The residue is taken up with 5 c.c. of water and a few drops of nitric acid, and the thorium precipitated by addition of 5 to 10 c.c. of solution I. From 10 to 20 c.c. of solution II. are added, and the mixture digested for fifteen minutes at 60° to 80° C. The thorium iodate is filtered off, washed with solution II. and then transferred to a beaker by means of a wash-bottle jet. It is decomposed by boiling with ammonia and the thorium hydroxide is filtered off, washed free from iodate, dried, and weighed as ThO_2. In the filtrate from the thorium iodate the scandium is precipitated by means of ammonia, and finally weighed as Sc_2O_3. G. C. J.

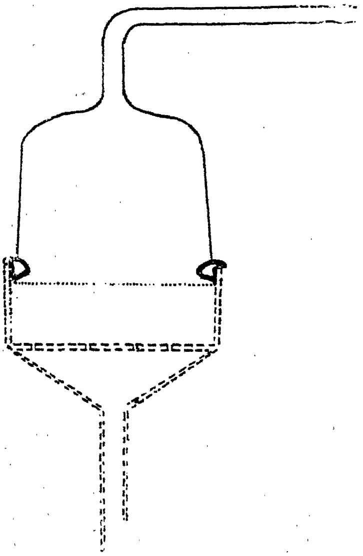
Analysis of Ferro-Vanadium and Estimation of Vanadium in Presence of Arsenic. W. Trautmann. (*Zeitsch. anal. Chem.*, 1911, **50**, 371-372.)—In presence of vanadic acid, arsenic acid is not reduced by sulphurous acid, and in hot strong sulphuric acid solution arsenious acid is instantly and quantitatively oxidised to arsenic acid by means of permanganate. The troublesome separation of arsenic preparatory to a vanadium titration is, therefore, unnecessary. The author fuses powdered ferro-vanadium with a mixture in equal proportions of sodium peroxide and carbonate, and extracts the melt with hot water and filters. The filter, with its contents, is dried, incinerated, and fused once more, and the product of the fusion extracted with water and the filtered solution added to the principal solution contained in a 2-litre flask. The solution is made strongly acid with sulphuric acid and heated to boiling, and a few drops of permanganate are added to oxidise any arsenic reduced by the hydrogen peroxide on acidifying. Reduction of the vanadium is effected by means of an aqueous solution of sulphurous acid, the excess of which is expelled by boiling whilst passing a current of carbon dioxide, and the vanadium is then titrated with permanganate as usual. G. C. J.

APPARATUS, ETC.

Some Forms of Glass Manometer. C. G. Jackson. (*J. Chem. Soc.*, 1911, **99**, 1066-1071.)—Three varieties of glass manometer, suitable for the measurement of pressures of vapours which attack mercury, or for measurements in which the experiment requires the manometer to be at a high temperature, are described and illustrated in the paper. The author has made use of one of these manometers in

studying the dissociation of cupric bromide, and the application of the manometer to such a purpose is explained, and the whole arrangement of apparatus is figured in the text.
G. C. J.

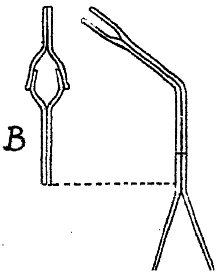
Addition to the Buchner Funnel. A. C. G. Egerton. (*Proc. Chem. Soc.*, 1911, **27**, 189-190.)—The apparatus consists simply of a small glass hood, which can be connected to a drying-tube. The glass hood is made of such a diameter that it fits inside a Buchner funnel, the joint being made air-tight with a rubber ring situated in a groove round the rim of the hood. It is well to employ a gentle pressure with the ring of a retort-stand to hold the hood in position, unless working with air under diminished pressure. A precipitate collected in the usual manner on the funnel can be dried by means of this simple additional apparatus in a very short time. Besides dispensing with the trouble of transferring the precipitate to a desiccator, the apparatus saves much time in the drying. The drying is most rapid when a steady, not too rapid, stream of air is drawn through the apparatus by means of the filter-pump after passing through a large drying-tower filled with calcium chloride; further, the hood, which is made of blown glass or, for very rapid drying, of metal, can be warmed with a flame. The precipitate can, if desired, be dried under diminished pressure by affixing a screw clip to the air inlet of the drying-tower. Another application of the apparatus is the drying of a precipitate in a non-reactive atmosphere; the gas can be circulated round and round, if necessary, by means of a blowing arrangement fitted to the filter-pump.

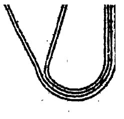


This small, inexpensive apparatus changes a Buchner funnel into an exceptionally efficient desiccator, which dries, not only precipitates collected on the funnel by the usual methods employed therewith, but also can be used conveniently for drying crucibles, etc., in a current of dry air.

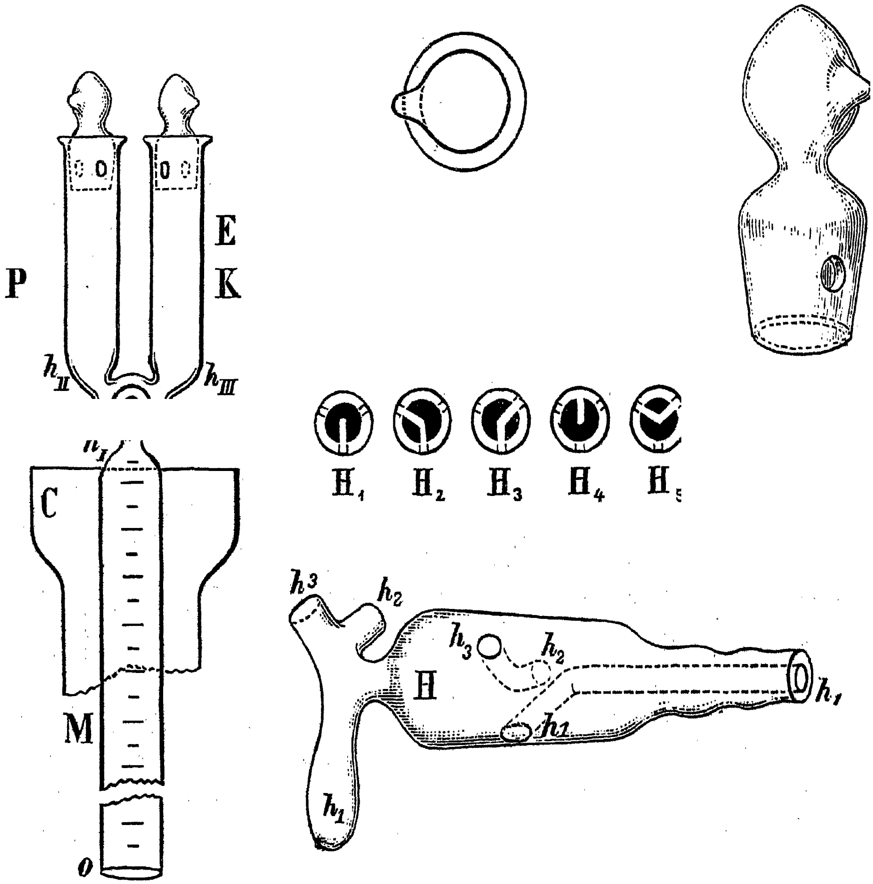
Melting-Point Determinations. G. A. Menge. (*U.S. Treasury Dept. Hygienic Lab. Bulletin*, 1911, 70, 1-101.)—A report of experiments carried out with the object of prescribing, in the ninth U.S. Pharmacopœia, the use of a simple method, so far rigidly defined, that it may be expected to yield approximately identical results in the hands of all operators. The apparatus finally recommended is a 10 by 3 cm. test-tube with walls about 1 mm. thick, and constructed of glass which will stand heating over an open flame. The test-tube is half filled with sulphuric acid, or for high temperature work, with a mixture of 70 parts by weight of sulphuric acid with 30 parts of potassium sulphate. The liquid is stirred continuously by a suitable (hand) stirrer. The substance to be examined is contained in a capillary tube not less than 0·8 mm. nor more than 1·25 mm. in internal diameter, this tube to contain a solid column of the substance 3 mm. high. The substance is heated rapidly to within 25° C. of its melting-point, then at the rate of 3° a minute until it begins to melt, as judged by the observation that some one point in the column of sample has collapsed against the side of the capillary. The rate of heating is then reduced to 0·5° a minute. A melting *interval* is to be returned, the beginning being that temperature at which some point in the column collapses, the end the temperature when the last trace of sample becomes liquid. All results should be corrected for the emergent stem of the thermometer in accordance with a prescribed formula. It is suggested that, if possible, some department of the U.S. Government should issue official thermometers of uniform construction. In that case a table or curve should be incorporated in the Pharmacopœia, showing the corrections to be made for the emergent stem. The need for uniformity of method in the determination of melting-points is illustrated by reference not only to foreign pharmacopœias, but to recently published and widely conflicting values which have been given for substances which are readily obtainable in a state of extreme purity, as judged by the shortness of the melting interval and other criteria. G. C. J.

Determination of the Density of Liquids. H. Hartley and W. H. Barrett. (*J. Chem. Soc.*, 1911, **99**, 1072-1074.)—Two forms of pyknometer were used, each made from test-tube glass, weighing about 5 grms. and holding 20 to 25 c.c. The form B, used for determining densities at temperatures below that of the laboratory, has a bulb to allow the liquid to expand, and a cap which should be ground on outside. The diameter of the capillary is 0·3 mm. The authors make use of a counterpoise made from the same kind of glass as the pyknometer, and having nearly the same surface area. By making the external volume of the counterpoise nearly equal (within 0·5 c.c.) to the sum of the volumes of the pyknometer and of the liquid it holds when full, the calculation of a series of densities may be greatly simplified without introducing an error greater than 2 units in the sixth decimal place of the density, even if the temperature should rise 10° C. and the barometer falls 30 mm. between the weighings of the pyknometer full of water and of the liquid to be tested. By the use of this apparatus and a thermostat constant to ±0·01° C., the authors were able to determine the densities of dilute (under 2 per cent.) aqueous solutions of several salts with an error not exceeding four parts in a million. In order to obtain this degree of accuracy in the case of liquids with a larger co-efficient of expansion, more accurate temperature regulation would be required. G. C. J.





Apparatus for Flue Gas Analysis. **A. Gawalowski.** (*Zeitsch. anal. Chem.* 911, **50**, 435-439.)—The apparatus consists of a 100 c.c. measuring-tube, surmounted y a three-way cock of special construction, and provided with two nitrometer cups ach of which is stoppered, the hollow stoppers having each a hole which may be nade to coincide with a hole in the ground seating. In the illustration one form of he three-way cock is shown on an enlarged scale; but it has been found better to make the plug of these cocks hollow and to fuse in small tubes rather than to bore channels in a solid plug. In use, the measuring-tube, which is open at the bottom, is sunk in a cylinder C of brine, the cock being in the position H_1 in which the measuring-tube is connected to the channel h_1, which allows air to escape. The end of the plug h_1 is now connected to the flue and the measuring-tube raised. The cock is turned to the position H_2 or H_3, and the first charge of gas blown off through either of the cups by sinking the tube in the brine cylinder. The tube is refilled with gas, its volume adjusted to exactly 100 c.c., and strong potash is admitted from the cup NP. After measuring the reduction in volume due to absorption of carbon dioxide, alkaline pyrogallol is added from the same cup NP and the oxygen estimated.



Finally, an ammoniacal solution of cuprous chloride is admitted from the other cup K to absorb carbon monoxide. As made now, the open end of the measuring-tube is somewhat constricted to facilitate closing by the finger, if it be desired to shake the contents of the tube. The maker is C. Glatzl, Mährisch-Ostrau. G. C. J.

Apparatus for the Maintenance of Constant Pressures Above and Below the Atmospheric Pressure. Application to Fractional Distillation. J. Wade and R. W. Merriman. (*J. Chem. Soc.*, 1911, **99**, 984-997.)—For pressures below that of the atmosphere, air is exhausted continuously from the system by an ordinary water-pump. For pressures above that of the atmosphere, air (or other gas) is forced in continuously by an automatic compressor, actuated by falling mercury. The pressure is kept constant by a regulator consisting of an open syphon manometer, which allows air to escape from or enter the system as soon as the desired excess or diminution of pressure is exceeded.

Devices are described in connection with fractional distillation, more especially for changing the receiver without disturbing the pressure. G. C. J.

REPORT.

Analysis of Potable Spirits, and its Bearing on the Definition of Whisky. E. A. Mann. (*Government Paper, Western Australia,* 1911.)—An analytical examination of 108 samples of whiskies of all kinds, together with a critical consideration of the figures placed before the English Royal Commission (1908), have convinced the author that it is quite possible to classify spirits by means of chemical analysis alone; thus it can be safely stated which are pure malt, pot still spirits, and which are blends containing at least 75, 50, or less than 50 per cent., of pot still spirit. A large number of very complete analyses are given, the Allen-Marquardt method being employed for determining the higher alcohols (*cf.* Mann and Stacey, ANALYST, 1907, **32**, 219, 258). The Report concludes with a concise description of the other methods employed, and photographs of special apparatus are included. A. R. T.

NOTE.—This is directly opposed to the commonly accepted opinion of this country.—EDITOR.

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REVIEWS.

LABORATORY TEXT-BOOK FOR BREWERS. By LAWRENCE BRIANT. Third edition. Edited by HAROLD HARMAN. Pp. 444. London, 1911. Published by Fell and Briant. Price 10s. 6d. net.

Those familiar with the first edition of this well-known textbook, published some twenty-five years ago, will remember it as practically the first attempt to describe analytical processes of use in a brewer's laboratory. In the edition now under review, the author and his editor have, wisely we think, adhered to the original plan of describing processes of a well tried and eminently practical kind. The textbook contains fifteen chapters, entitled as follows :

I. Apparatus and Manipulation. II. Part I.: Nitrogenous Bodies or Proteins; Part II.: Enzymes; Part III.: Carbohydrates. III. Malt Analysis and Interpretations; Part I.: White Malt; Part II.: Coloured Malt; Part III.: Barley. IV. Unmalted Grain. V. Sugars. VI. Caramel. VII. Hops. VIII. Water Analysis. IX. Sulphites. X. Original Gravity of Beer. XI. Complete Analysis of Malt Wort and Beer. XII. Estimation of Arsenic in Brewing Materials. XIII. Microscopical Work. XIV. The Polarimeter. XV. Standard Solutions and Reagents.

In all cases where the method of analysis of a brewing material is described an actual example is given, together with interpretations, the latter being of an essentially conservative kind. While, however, courses other than safe and sound ones, have been avoided, we must confess to some sense of disappointment that an advisory chemist of Mr. Briant's experience still views a question like that of the influence of wort nitrogen from grounds which seem largely speculative. In spite of all that has been said in a general way about the influence of the "soluble uncoagulable albuminoids" on the stability of the finished beer, we think we can still deny the existence of the experimental evidence by which these factors have been connoted. We can quite agree that a high specific gravity for the cold-water extract of malt indicates an unsuitable material, but beyond this we still require direct proof that it is the nitrogenous constituents of the extract which influence the stability of beer, and, until such proof is forthcoming, all this talk about good and bad nitrogen will remain unconvincing. Our sceptical outlook is not intended to cast doubt on the value of this book, which, however, contains some matter open to criticism—*e.g.*, we do not like the author's procedure by which he standardises Fehling's solution for the volumetric estimation of sugar. Surely in these days, when pure chemicals (the author specifies them) are readily procured, Fehling's solution can be made up as a standard solution? And, in any event, we do not believe that it can be accurately standardised by an invert sugar solution prepared according to the author's description.

We also question whether the free sulphur in hops can be determined by passing "a current of hydrogen through a decoction of hops, when, if present, the sulphur will unite with that gas to form sulphuretted hydrogen"; in fact, this method is generally known to be worthless for quantitative purposes.

There are minor blemishes in the book which call for some notice. Thus proteins soluble in alcohol are not "protamines," the name of a totally different class, but, if the American term be used, "prolamins." Other errors which may prove very irritating, especially to the novice, concern some of the physical constants quoted. The optical constant of maltose is given (p. 93) as $[\alpha]_{D3\cdot86}+138$, which is not correct; and in the footnote on the same page we find the sign $[\alpha]_{D17\cdot5}$ without any explanation that 17·5 refers to the temperature of observation. Similar mistakes occur in describing other sugars—*e.g.*, on p. 211. The erroneous curve for invert sugar in the diagram on p. 102 seems in danger of perpetuation, and we take the opportunity of pointing out that, instead of as shown, the curve should lie at all points as the mean of the curves for glucose and lævulose.

For those brewers who wish their laboratory run on safe and sound, rather than highly scientific lines, we think Mr. Briant's book fully justifies the title he has given to it.

RUBBER. By CLAYTON BEADLE and H. P. STEVENS, M.A., Ph.D., F.I.C. (Pitman's Common Commodities of Commerce.) 1911. London: Sir Isaac Pitman and Sons, Ltd. Price 1s. 6d. net.

This booklet is one of a series on "Common Commodities of Commerce," and its object, according to the authors' prefatory note, is to supply an account of the production of the raw material, and the subsequent manufacturing processes, which will be found suitable to the requirements of the average reader who is desirous of obtaining general information on the subject, to the planter who wishes to know something of the manufacturing side of the industry, and to the manufacturer who is interested in the preparation of his chief raw material. The little work is brightly written and eminently readable, and contains a number of illustrations of rubber-trees and manufacturing machinery. The coloured frontispiece, which is from a water-colour painting by Dr. Stevens, affords an excellent idea of the appearance of plantation *hevea*-trees. The book is obviously not intended for the analyst or scientific worker, and it would, therefore, be unfair to criticise it from this point of view. P. SCHIDROWITZ.

NATIONAL PHYSICAL LABORATORY. Collected Researches. Vol. VII. 1911. Pp. 228 + iv.

This volume includes the records of eight different researches, more than half the total space being occupied with an account of Rosenhain and Lantsberry's researches on the properties of some alloys of copper, aluminium, and manganese, to which is appended an interesting note on the corrosion of alloys of copper and aluminium when exposed to the sea. The results of this work have appeared already in the Proceedings of the Institution of Mechanical Engineers, and it should be said that there is nothing in this volume which has not already appeared elsewhere; but the files of six different journals would have to be searched to find the eight papers here collected together. Dr. Kaye's work on the expansion and thermal hysteresis of fused silica will be of interest to many chemists; and Paterson and Rayner's experiments on comparative life-tests of glow lamps will be of interest to a smaller number. The object of these latter experiments was to determine the agreement which it is possible to obtain when life-tests are carried out on glow lamps by different laboratories. So far as the tests go, they indicate that measurements of the useful life of carbon filament lamps can be carried out on the basis of the Engineering Standards Committee's specification to give results within an accuracy of ± 5 per cent. From an experimental point of view, tests on metallic filament lamps should yield a higher accuracy. G. C. JONES.

NATIONAL PHYSICAL LABORATORY. Report for the year 1910. Pp. 102, with four plates.

The first nine pages are occupied with the Report of the Executive Committee, which reviews briefly the whole of the work accomplished during the past year, and a statement of work proposed for the year 1911 fills another seven pages. These are followed by the Report of the Director on the Physics Department, and the Reports to the Director by the Superintendents of the Engineering Department, the Department of Metallurgy and Metallurgical Chemistry, and of the Richmond Observatory. The Report of the Advisory Committee for the National Experimental Tank is also included.

Of special interest to chemists is the report (pp. 45-52) of a research which had for its object to determine the cause of the higher readings given by the Abel-Pensky flash-point apparatus (particularly the German form of this apparatus), as compared with those yielded by the ordinary Abel tester. The true cause of the differences has almost certainly been ascertained, and a detailed report to the Petroleum Commission is promised. The results of work carried out at the National Physical Laboratory are communicated to the particular learned society they are most likely to interest, but as the domains of the societies overlap, it is not always easy to find a particular paper which one may know to have issued from Teddington. The present report meets this difficulty by including a list of all papers (official or unofficial) read during the years 1909 and 1910 by members of the staff of the National Physical Laboratory. G. C. Jones.

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INSTITUTE OF CHEMISTRY.

Pass List: June—July Examinations, 1911.

(Examinations were held in London and Glasgow.)

Of thirty-one candidates who presented themselves for the Intermediate Examination, the following seventeen passed: J. L. S. Allen, K. C. G. Arbuthnot, B.A., R. Clark, T. W. Derrington, E. C. Evans, B.Sc., H. Gilmour, R. E. Griffiths, B.Sc., C. L. Hinton, R. P. Keith, J. G. King, R. Linch, B. B. Murdoch, M.A., B.Sc., M. J. Robb, B.Sc., J. W. Tait, M.A., F. G. Tarn, T. A. Wilson, F. G. C. Walker. Of thirty-four candidates who presented themselves for the Final Examination for the Associateship, twenty-three passed. In the Branch of Mineral Chemistry: G. Hamilton, S. R. Illingworth, A.R.C.S., E. A. Masters, B.Sc., C. B. Ross, B.Sc., J. Sorley, A. F. Suggett; in the Branch of Physical Chemistry: Arthur Bramley, B.Sc., A.R.C.S.; in the Branch of Organic Chemistry: E. Anderson, O. L. Brady, B.A., D. S. Dawson, B.Sc., W. Jewell, J. A. McRae, M.A., W. G. Prescott, S. P. Schotz, D. B. Steinberg, B.Sc., J. C. White, L. Wilson, B.Sc.; and in the Branch of the Chemistry of Food and Drugs, Fertilisers and Feeding Stuffs, Soils and Water: H. J. Evans, Miss M. Gazdar, B.Sc., H. Lowe, B.Sc., W. R. Pratt, H. B. Stevens, J. A. L. Sutcliffe. Of four candidates examined for the Fellowship, two passed in the Chemistry of Food and Drugs, etc.: J. Hawthorne, B.A., Ph.D., and A. Miller, M.R.C.S., Major I.M.S.; and one a Special Examination in General Chemistry: G. H. Stanley, A.R.S.M. One Candidate for the Intermediate Examination presented himself at Singapore in June, and passed: A. M. Bailey. The Board of Examiners reported on seventy candidates in all, and of these forty-four passed.

SEPTEMBER, 1911. Vol. XXXVI., No. 426.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE EFFECT OF FEEDING WITH COCOANUT CAKE AND LINSEED CAKE ON THE COMPOSITION OF BUTTER-FAT.

By H. T. CRANFIELD.

WHILST there is a considerable difference of opinion as to whether feeding influences the composition of milk or not—and many experiments have been carried out with this object in view—little investigation seems to have been made in this country concerning the influence of feeding on the composition of butter-fat. In Germany, however, some work has been carried out by H. Lührig (*Zeitsch. Untersuch. Nahr. Genussm.*, 1906, **11**, 11), which deserves notice in connection with the present communication. Lührig watched the effect of feeding with cocoanut cake on the composition of the butter, and found that there was a considerable rise in the Polenske number, and but little effect on the Reichert-Meissl number. In consequence of this variation he recommended an increase of 0·8 in the limits adopted by Polenske for genuine butters in his original paper in 1904.

At the present time cocoanut cake is being placed on the English market in much larger quantities than in previous years; and since it is not unlikely that this food will be used more extensively by dairy farmers in the near future, I decided to take advantage of an experiment on the feeding of dairy cows which was being carried out during the spring of this year at the Midland Agricultural and Dairy College.

DETAILS OF THE EXPERIMENT.

Two sets (A and B) of four cows* each were employed, each cow receiving the following ration daily: 56 pounds mangels, 7 pounds oat-straw chaff, 2 pounds mixed meal (bran, dried grains, rice meal), and 14 pounds hay. In addition to this ration, all the cows received during the first week 5 pounds of undecorticated cotton cake daily. In the second and third weeks Set A received 5 pounds of cocoanut cake, and Set B 4½ pounds of linseed cake. These foods were reversed in the fourth and fifth weeks, and for the last week all the cows reverted to cotton cake.

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* All the cows were cross-bred shorthorns.

Three times a week about half a pint of cream was reserved—a consecutive night and morning—from the milk of each set of cows. The cream was ripened with a starter, and churned after twenty-four hours in a small glass experimental churn. Once a week the whole of the milk from a consecutive night's and morning's milking was separated, and the cream (corresponding to one of the small samples) churned in an ordinary barrel churn. A sample from this bulk of butter was taken for analysis, to serve as a control in the method of churning and also in the analytical determinations.

The samples of butter were melted at a low temperature, and filtered in the ordinary way. The refractometer figure, Reichert-Meissl number, Polenske number, and Shrewsbury and Knapp figure, were determined. The results are recorded in the accompanying table:

Number of Sample.		$[N]_D$ 35° C.		Reichert-Meissl Number.		Polenske Number.		Shrewsbury and Knapp Figure.	
		Set A.	Set B.	Set A.	Set B.	Set A.	Set B.	Set A.	Set B.
		Cotton.	Cotton.	Cotton.	Cotton.	Cotton.	Cotton.	Cotton.	Cotton.
1st week.	1	46·5	46·5	30·7	31·4	2·3	2·2	27·2	25·6
	2	46·0	46·5	30·7	31·1	2·3	2·0	28·7	27·3
	Bulk 2	46·0	46·5	30·7	31·1	2·3	2·0	—	—
		Cocoanut.	Linseed.	Cocoanut.	Linseed.	Cocoanut.	Linseed.	Cocoanut.	Linseed.
2nd week.	3	46·0	48·0	29·9	27·7	2·6	1·5	28·9	24·7
	4	45·0	48·0	29·6	28·8	2·9	1·7	32·9	25·5
	5	44·5	48·0	30·3	29·5	3·3	1·7	34·3	27·0
	Bulk 5	44·5	47·5	29·8	29·4	3·3	1·8	—	—
3rd week.	6	44·5	47·5	28·9	28·8	3·4	1·7	34·7	25·7
	7	44·0	47·5	29·4	28·7	3·4	1·7	40·1	29·4
	8	43·0	47·5	29·5	29·6	3·7	1·8	37·6	25·7
	Bulk 8	44·0	47·5	28·7	29·4	3·4	1·8	—	—
		Linseed.	Cocoanut.	Linseed.	Cocoanut.	Linseed.	Cocoanut.	Linseed.	Cocoanut.
4th week.	9	45·0	46·5	28·3	30·2	2·8	2·5	34·4	28·4
	10	46·0	46·0	28·7	28·4	2·5	2·3	—	—
	11	45·0	46·5	29·8	29·2	2·8	2·3	—	—
	Bulk 11	46·5	45·0	28·9	29·4	2·3	2·8	—	—
5th week.	12	47·0	45·0	28·8	30·0	2·2	2·8	—	—
	13	47·0	45·5	29·4	29·9	2·5	2·9	—	—
	14	46·5	44·5	29·5	31·7	2·3	3·1	—	—
	Bulk 14	46·5	44·5	29·1	31·7	2·3	3·0	—	—
		Cotton.	Cotton.	Cotton.	Cotton.	Cotton.	Cotton.	Cotton.	Cotton.
6th week.	15	47·0	45·0	28·4	29·8	2·2	2·7	—	—
	16	46·5	47·5	30·3	29·1	2·5	2·0	—	—
	17	46·0	46·5	30·8	32·1	2·7	2·3	—	—
	Bulk 17	46·0	46·0	30·7	31·8	2·7	2·3	—	—

Bell, W. J., Lloyd, C. F., *and* Robinson, R. A., The Sale of Food and Drugs Acts, *Review*, 325
Bellmer, E., Estimation of the Specific Gravity of Oils, 557
Benedict, S. R., Estimation of Reducing Sugars, 298
Beneker, J. C., Rapid and Accurate Method for the Analysis of White Metals, 606
Benner, R. C., *and* Hartmann, M. L., Rapid Determinations and Separations by Means of the Mercury Cathode and Stationary Anode, 83
————, *and* Ross, W. H., Rapid Determination of Nickel and Cobalt by Means of the Gauze Cathode and Stationary Anode, 307
————————, Rapid Determination of Silver and Cadmium by Means of the Gauze Cathode and Stationary Anode, 473
Benson, H. K., *and* Pollock, C., New Accelerated Test for Paints, 608
Berget, A., Estimation of the Salinity of Sea Water by Means of the Refractometer, 311
Bernhardt, P., New Indicator for the Estimation of "Titratable" Alkali in Blood, 415
Bernier, R., *and* Péron, G., Estimation of Small Quantities of Iodides in the Presence of Other Compounds, 241
Bertrand, G., Estimation of Small Quantities of Manganese, Especially in the Ash of Plants or of Animal Organs, 369
Besson, A. A., Estimation of the Doughing Value of Flour, 216
————, The Anhydride of Lactic Acid, 235
————, Valuation of Tea, 454
Beutell, A., Mercury Pump for Rapidly Obtaining a High Vacuum, 89
Bevan, E. J., Note on the Gravimetric Estimation of Phosphorus in Milk, 583
Beythien, A., Hempel, H., Simmich, P., Schwert, W., *and* Wiesemann, C., Estimation of Formaldehyde by the Ammonia Method, 421
Bird, W. H., Collective Index of the Journal of the Institute of Brewing, 1887-1910, *Review*, 576
Bjorksten, R., *and* Nyman, M., Precipitation of Cocaine Solutions with Platinum Chloride, 354
Black, J. W., *and* Cockburn, T., The Estimation of Quinine as the Acid Citrate in Certain Organic Liquids, 396
Blair, H., Estimation of Sulphur in Coal-Gas and of Ammonium Sulphate, 309
Blakeley, A. G., *and* Chance, E. M., Accurate Technical Estimation of Calorific Value of Anthracite, 518
————————, Estimation of Lead in Alloys Containing Antimony and Tin, 367
Blarez, C., Analysis of Turpentine Oil, 546
Blücher, H., Modern Industrial Chemistry, *Review*, 325
Blum, W., *and* Hillebrand, W. F., Estimation of Manganese by the Bismuth Method, 439
Blume, F., Holder for Classen Dish, 180
Bockermann, W., Estimation of Soap in Heavy Grease, 559
Böddener, K. H., *and* Tollens, B., Modification of the Furfural Method for the Estimation of Pentosans, 161
Bodmer, —, Note on Gorgonzola Cheese, 63
Bödtker, E., Estimation of Free Fatty Acids in Fats, 355
Bogoluboff, P., *and* Wdowiszewski, H., Estimation of Chromium in Chrome Tungsten Steel, 83
Bokorny, T., Microchemical Detection of Protein, 111
Bolton, E. R., *and* Revis, C., Further Analysis of Ghee, 392
————————, Observations on Some Methods of Estimating Cocoanut Oil and Butter in Butter and Margarine, 333

Bordas, F., *and* Touplain, F., Distribution of the Phosphoric Acid in Milk, 345

Bos, J. L. M. van der Horn van den, Quantitative Separation of Barium, Strontium, and Calcium, 364

Bourion, F., General Method for the Preparation of Anhydrous Chlorides, and its Application in Chemical Analysis, 31

Bouyssy, M., *and* Henriet, H., Method of Measuring the Degree of Vitiation of the Atmosphere, 363

Bowser, L. T., Detection of Small Quantities of Potassium as the Cobalti-Nitrite, 606

——————, Estimation of Very Small Amounts of Calcium by Means of Permanganate, 174

——————, Titrimetric Estimation of Phosphorus in Small Amounts, 246

Boyle, J. J., Comparison of Methods for Determination of Nickel in Steel, 524

Boynton, C. N., *and* Gooch, F. A., Separation and Estimation of Barium in Presence of Calcium and Magnesium by the Action of Acetyl Chloride and Acetone on the Mixed Chlorides, 426

Brady, W., *and* Touzalin, L. A., Determination of Dust in Blast Furnace Gas, 601

Bressanin, G., Volumetric Estimation of Mercuric Chloride, 34

Bresson, M., *and* Achalme, P., Influence of the Viscosity on the Action of Invertase According to the Concentration of Saccharose, 416

——————————————, Method for Ascertaining whether a Liquid Contains One or More Enzymes, 110

Briant, L., Laboratory Textbook for Brewers, *Review*, 441

Brinton, P. H. M. P., Estimation of Manganese by the Bismuthate Method, 305

——————————, *and* Fresenius,, H., Estimation of Potassium as Potassium Platini-Chloride, 178

Brouvert, E., Volumetric Determination of Nitric Acid in Nitrocellulose, Nitroglycerol, and Other Compounds, 34

Brown, A. J., *and* Ward, G. B., Valuation of the Antiseptic Properties of Hops, 65

————, C. E., *and* Shadinger, G. B., Flask for Distillation of Tar, 529

Browne, C. A., Normal Weight of Dextrose, 78

——————, Use of Temperature Corrections in the Polarisation of Raw Sugars and Other Products upon Quartz Wedge Saccharimeters, 297

——————, *and* Bryan, A. H., Analysis of Commercial Dextrins, 276

Brugemann, —, Estimation of Fillers (Mineral Matter) in Rubber Mixtures, 558

Brumwell, H., Microscopical Examination of Gambier, 344

Brunck, O., Estimation of Hydrogen in Gas Mixtures, 84

Brunetti, W., Changes which Mercury Compounds Undergo when in Contact with Organic Matters, 154

Brünnich, J. C., *and* Smith, F., Detection and Estimation of Arsenic Acid in Presence of Arsenious Acid by Means of Magnesia Mixture, 30

Bryan, A. H., *and* Browne, C. A., Analysis of Commercial Dextrins, 276

——————, Given, A., *and* Straughn, M. N., Estimation of Sugars in Feeding-Stuffs, 453

Buchanan, G. S., On the Work of Inspectors of Foods, 128

Buchner, G., Analysis of Beeswax, with Special Reference to East Indian and the So-called China Beeswax, 554

Buchta, F., *and* Oettinger, C., Soya Bean Oil, 358

Buckmaster, G. A., *and* Gardner, J. A., Composition of the Gases of the Blood in Chloroform Anæsthesia, 154

Buckminster, I. H., *and* Smith, E. F., Electrolytic Separations, 32

Budde, J., Estimation of Caoutchouc as Tetrabromide, 159

——————————, Fatty Acids in Soaps, 355

Burgess, W. J., The Determination of the Amount of Dissolved Oxygen absorbed by

Averages.—In compiling these averages, the transitory samples have been omitted—viz., Nos. 3, 9, 10, and 15:

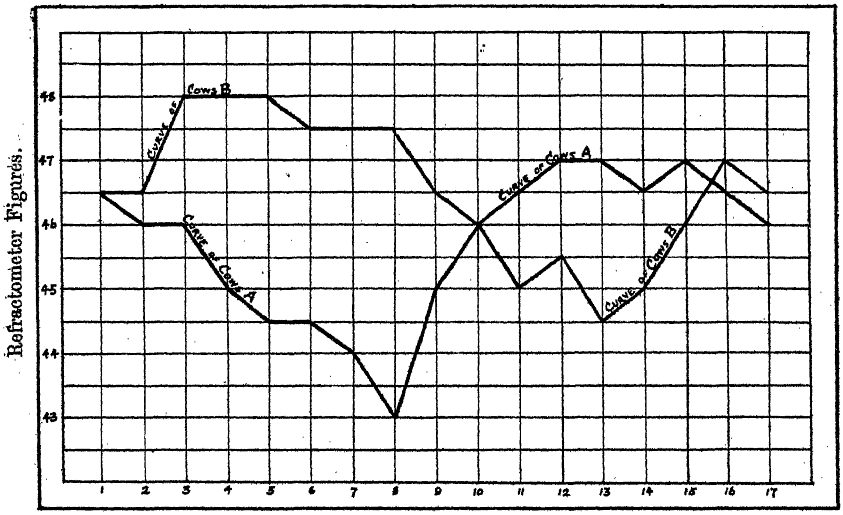
		$[N]_{D\,35^\circ}$ C.	Reichert-Meissl Number.	Polenske Number.
Cocoanut cake	A ...	44·2	29·5	3·3
	B ...	45·4	30·2	2·9
Linseed cake	A ...	46·2	29·1	2·3
	B ...	47·7	29·1	1·7
Cotton cake	...	46·4	30·8	2·3

It will be seen from this table that the figures given by the bulk samples of butter agree very closely, and in many cases are identical, with the corresponding small samples of butter.

Refractometer Figure.—On perusing the foregoing table, it will be noticed that the two cakes have had a marked influence on this figure. The linseed cake caused a distinct rise in the refractive index, while the cocoanut cake gave a reverse variation. The rise and fall of this figure can be easily seen in the accompanying curves:

REFRACTOMETER CURVES (35° C.).

A & B Cotton. 1st Week. A Cocoanut. B Linseed. 2nd & 3rd Weeks. A Linseed. B Cocoanut. 4th and 5th Weeks. A & B Cotton. 6th Week.



Number of Sample.

Reichert-Meissl Number.—The influence of the feeding on this number is not so great as in the case of the refractometer figure. Certainly some slight variation seems apparent—viz., a falling in the case of the linseed cake, and a rise due to the cocoanut cake. Both cakes, however, give a lower average figure than the cotton cake.

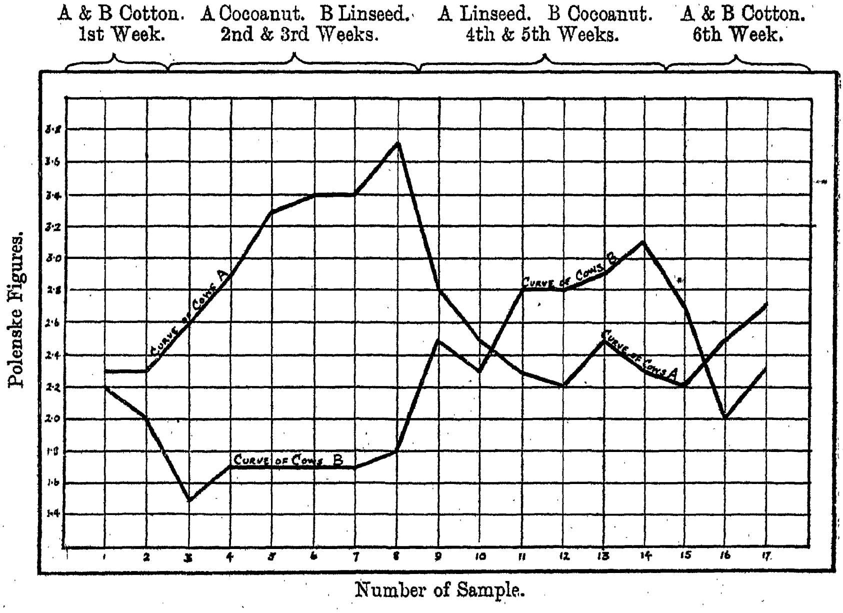
Polenske Number.—On this number the feeding has had a most remarkable influence, the linseed cake causing the figure to fall to a very low point, while the cocoanut cake raised the number considerably.

Taking the figures obtained from Samples 8 A and 8 B, we get the following comparison:

	8 A.	8 B.
Reichert-Meissl number	29·5	29·6
Polenske number	3·7	1·8

The curve given below shows very closely the variations in this figure during the experiment:

POLENSKE CURVES.



Polenske in original paper (1904) compares the Reichert-Meissl and Polenske numbers obtained for normal butters, and gives the highest figures allowable for genuine butter. In this experiment several samples gave figures outside Polenske's limits. For example—

Number of Sample.	Reichert-Meissl Figure.	Polenske Figure.	Polenske Highest Allowable Figure.
6 A	28·9	3·4	3·0
8 A	29·5	3·7	3·5
Bulk 8 A	28·7	3·4	3·0

Lührig (*loc. cit.*) quotes even higher figures than these, but the amount of cocoanut cake fed in his experiment was up to 6 kgrms. daily—a quantity much in excess of that fed during this experiment. In view of the likelihood of the increased use of cocoanut cake in this country for feeding dairy cattle, this influence is of importance to the butter-producer.

Shrewsbury and Knapp Figure (ANALYST, 1910, **35**, 36).—The method for this estimation has only recently been brought to the notice of analytical chemists, and with a view to testing the method, I made a few determinations, obtaining the results given in the previous table. All the results given are the mean of at least two determinations, and where the variation exceeded 0·5 a third estimation was made. Shrewsbury and Knapp mention a limit of 32 for their figure for genuine butter, but it will be seen from my figures that this limit has been exceeded in six cases, one giving as high a figure as 40·1, which, according to Shrewsbury and Knapp, would represent over 10 per cent. added cocoanut oil. It seems, therefore, that this method does not show any superiority over the Polenske method, though it is certainly a more expeditious estimation to carry out.

Rancidity.—The cakes appeared to have a different effect on the keeping properties of the butter-fat. The samples were all kept in small corked bottles or flasks, and it was noticed after several weeks that the butters from the linseed and cotton cakes had turned quite white and rancid, while those from the cocoanut cake showed only a slight change. This preservative effect is undoubtedly an advantage in favour of cocoanut-cake-fed butter.

Quality of the Butter.—The following report was given on the quality of the weekly samples of butter in bulk, and for this report I am indebted to Mr. Alec Todd, Instructor in Dairying at the Midland Agricultural and Dairy College:

"No very great differences were shown in the texture and flavour of the two sets of butters. The butter from the cocoanut cake, however, had as a rule a firmer and less oily texture and better flavour than the linseed-cake butter."

CONCLUSIONS.

1. It is apparent from these results that feeding has a great influence on the composition of butter-fat.

2. The feeding of cocoanut cake to dairy cows produced a butter which gave a refractometer figure below the normal, while the Polenske number was very high. The Reichert-Meissl number was, however, quite normal.

3. Feeding with linseed cake influenced the butter in respect of the Polenske number, butters from this source giving a figure much below the normal. The refractometer figure was also influenced, a distinct rise being noticeable.

4. Cocoanut cake produced a butter of good quality, possessing better keeping properties than butter from linseed or cotton cakes.

5. The Shrewsbury and Knapp method for detection of cocoanut oil in butter possesses practically no advantages over the Polenske method.

MIDLAND AGRICULTURAL AND DAIRY COLLEGE,
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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Assay of Aconite. A. B. Stevens. (*Bulletin of Pharmacy*, June, 1911 through *Pharm. J.*, 1911, **87**, 34.)—The United States Pharmacopœia method, which is practically that of the author, is more rapid if carried out as originally devised; that is to say, the evaporation at 60° C. is performed in a flat-bottom evaporating-dish, and 5 grms. pumice powder are added to the percolate before evaporation. In this way the subsequent solution and filtration are much hastened.

The deterioration of aconite by age or moisture can be detected by both chemical and physiological methods of assay. Thus, a sample of aconite, showing 0·837 per cent. of alkaloids on assay, and giving the characteristic tingling sensation in a dilution of 1 : 1,200, gave the following results after the treatments specified:

	Alkaloids found per Cent.	Physiological Test.
1. Moistened with water and kept for several days in stoppered bottle with cotton moistened with chloroform	0·67 ; 0·66 ...	1 : 700
2. Moistened and kept in warm place till dense mould formed	0·30 ; 0·32 ...	1 : 450

Experiments by F. O. Taylor (*J. Ind. and Eng. Chem.*, 1909, **1**, 549) have shown that by the action of heat aconite preparations may be reduced almost to a physiologically inactive condition, and yet the chemical method of assay would not alone serve to detect this deterioration. Moreover, so far from the decomposition products causing the titration method to yield high results, in most cases the results were lower than by the gravimetric assay. Ordinarily, therefore, the deterioration of the drug may be chemically determined; and if due to heat, it may be detected by volumetric assay, since the weight of the ether-soluble residue is increased and its basic properties decreased. Aconite contains considerable quantities of chloroform-soluble bases (insoluble in ether) which are quite inactive physiologically. Thus, chloroform should not be used for the assay of aconite. The Squibb method of determining the largest dilution at which the characteristic sensation on the tongue may be produced is useful when applied by a sensitive person, but the results are very variable with different persons.

A. R. T.

Composition of Sweet Apricot Kernels. L. Rosenthaler and W. Schaeffer. (*Pharm. Zentralb.*, 1911, **52**, 507-508; through *Chem. Zentralbl.*, 1911, II., 35.)—The uthors have examined specimens of edible apricot kernels obtained from the South f France; the kernels had a pleasant, almond-like taste quite different from that of he bitter kernels imported from Japan. Their composition was as follows: Water, ·33; oil, 53·40; crude fibre, 4·76; ash, 2·60; total soluble matter, 27·87; reducing ugars, 8·08; reducing sugars after hydrolysis, 11·64; and proteins, 31·4 per cent. he oil expressed from the kernels had the following physical and chemical properties: Sp. gr. at 15° C., 0·9182; $[N]_{D\,25^\circ\,C.}$, 64·7; solidifying-point, 14·5° C.; iodine value, 95·03; saponification value, 187·76; Reichert-Meissl value, 0·96; insoluble fatty acids, 73·48 per cent. The oil was optically inactive. The fatty acids gave the following values: Solidifying-point, 5·1° C.; $[N]_{D\,25^\circ\,C.}$, 53·7; iodine value, 98·41; mean molecular weight, 290·55. The oil yielded negative results with Halphen's, Baudouin's, Soltsien's, and Becchi's tests, but gave a blue-violet coloration with Bellier's test, the coloration developing gradually and fading in a few minutes. The elaidin test yielded a viscous mass. W. P. S.

Detection of Cocoanut Oil in Butter by Means of Ewer's Method. E. Nockmann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 754-757.)—Results of analyses are recorded which show that when cows are fed on beetroot leaves the composition of the butter obtained from the cows is so altered that the figures obtained by Ewer's method (ANALYST, 1910, **35**, 353) are such as would indicate the presence of cocoanut oil in the butter. Of seven samples of butter obtained from cows fed on beetroot leaves, five gave "difference figures" varying from 7·8 to 8·9, the average figure for these butters before the period during which the cows were fed on the beetroot leaves being 11·5. It is also pointed out that the presence of 10 per cent. or more of cocoanut oil in butter can be detected by the usual analytical methods (*cf.* ANALYST, 1911, 404). W. P. S.

Detection of Cocoanut Oil in Butter by Means of Fendler's Method. A. Hepner. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 758-760.)—When examined by the ester method described by Fendler (ANALYST, 1910, **35**, 355), the mixed butter of a herd of cows gave a distillation figure of 4·7; but after the cows had been fed for some time on beetroot leaves this value rose to 7·5 (average), and on certain dates was over 9·0. The author considers, therefore, that the method possesses no advantage over the usual analytical processes for the detection of cocoanut oil in butter (*cf.* preceding abstract). W. P. S.

Examination of Honey by Fiehe's Reaction. H. Lührig and A. Scholz. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 721-741.)—Fiehe's reaction (ANALYST, 1908, **33**, 397) depends on the detection of hydroxymethylfurfural, traces of which are produced by the action of mineral acids on sucrose during the inversion of the latter in the preparation of artificial honey (invert sugar). As honey contains both sucrose and organic acids, it might be assumed that, when pure honey is heated, the sucrose is inverted, and that such honey would yield a positive reaction with Fiehe's reagent. A large number of experiments are recorded by the author; in these experiments sucrose and honey solutions were heated to 80° C. for thirty minutes or longer after the addition of varying proportions of different organic acids, and then tested with the reagent in the usual way. It was found, however, that there is a minimum concentration for each organic acid below which the reaction is not obtained. For instance, the sucrose or honey solution must contain not less than 0·1 per cent. of oxalic, or 2 per cent. of lactic, acetic, formic, butyric, or salicylic acids, before a positive reaction is obtained with the reagent. The small quantities of organic acids present in natural honey do not cause the latter to give a reaction with Fiehe's reagent, even when the honey has been heated for one hour at a temperature of 100° C. W. P. S.

Estimation of Nicotine in the Presence of Pyridine Bases. L. Surre. (*Ann. Falsif.*, 1911, 4, 331-334.)—The method proposed depends on the fact that nicotine has an optical rotation of −161·55° (200 mm. tube at a temperature of 20° C.), whilst pyridine and similar bases are optically inactive. The author finds that, for solutions containing from 1 to 6 per cent. of nicotine, the rotation is exactly proportional to the concentration, and that the presence of pyridine does not modify the rotation. Fifty c.c. of the sample are placed in a flask, together with 1 grm. of calcined magnesia and 2 grms. of powdered pumice, and distilled. When about 25 c.c. of distillate have come over, water is admitted to the flask drop by drop from a tapped funnel (the addition of the water can be regulated so as not to interfere seriously with the boiling of the contents of the flask), and the distillation is continued until 150 c.c. of distillate have been collected. This quantity will contain the whole of the pyridine present and also the greater part of the nicotine, and the amount of the latter is then estimated polarimetrically. Let A be the reading observed (angular degrees); then (for 50 c.c. of the sample taken and 150 c.c. of distillate) the amount of nicotine in grms. per litre of the sample will be $A \times 18{\cdot}57$. In order to obtain the whole of the nicotine, the contents of the flask are further distilled, with the gradual addition of water, until a second quantity of 150 c.c. of distillate have been received; and this distillate, containing only nicotine, is titrated with $\frac{N}{10}$ sulphuric acid, using luteolin as indicator; each c.c. of $\frac{N}{10}$ acid is equivalent to 0·0162 grm. of nicotine. As the quantity of nicotine in the second distillate is very small, the titration is more trustworthy than the polarimetric estimation. The method yields satisfactory results, the error not exceeding 0·7 grm. of nicotine per litre. W. P. S.

New Method for the Detection of Salvarsan. J. Abelin. (*Münch. med. Wochenschr.*, 1910, 58, 1002-1003; through *Chem. Zentralbl.*, 1911, II., 55.)—The est depends upon the formation of a bright red colour when diazotised dihydroxyiamino-arsenobenzene (salvarsan) is treated with alkaline resorcinol solution. A mall quantity of salvarsan is dissolved in about 3 c.c. of water, 4 drops of dilute ydrochloric acid are added, and the mixture is treated with a 0·5 per cent. sodium itrite solution, the mixture being cooled during the addition. The diazotised substance exhibits a greenish-yellow fluorescence, and when the solution is added drop y drop to a 10 per cent. resorcinol solution rendered alkaline with sodium hydroxide, red coloration is produced. Phloroglucinol, α-naphthol, etc., may be used in place resorcinol, but the latter gives the best results. The test may be applied directly o the urine of persons who have been taking salvarsan, as the constituents of the rine are without influence on the reaction. The test will detect the presence of lvarsan in the urine of a patient two hours after the substance has been injected bcutaneously. Atoxyl yields an orange coloration when it is diazotised and eated with resorcinol. W. P. S.

Estimation of Sugars in Feeding-Stuffs. A. H. Bryan, A. Given, and M. N. Straughn. (*J. Ind. and Eng. Chem.*, 1911, **3**, 486-492.)—The authors have compared two methods in general use—namely, extraction with (1) hot diluted alcohol (50 per cent.); and (2) cold sodium carbonate solution. The action of alcohol at the temperature employed, and of the sodium carbonate, in killing the enzymes or preventing their action, is an important factor in these methods, since by the activity of these ferments sugar is produced from the starch present in the products under examination, thus seriously affecting the results. The methods are best carried out as follows:

Alcohol Digestion Method.—One hundred and fifty c.c. of alcohol (50 per cent. by volume) and 12 grms. of the finely-ground material in a 300 c.c. graduated flask, are kept on the water-bath for one hour, the vapour from the flask being condensed by placing a small funnel in the neck. If the material be acid, 1 to 3 grms. of calcium carbonate should first be added. The contents of the flask are cooled and, if desired, allowed to stand overnight, and made up to 300 c.c. volume by the addition of neutral alcohol (95 per cent.). After thorough mixing, 200 c.c. of the supernatant liquid are evaporated at 100° C. to about 20 to 30 c.c. Sufficient saturated neutral lead acetate solution is added to produce a flocculent precipitate, and, after standing, the volume is made up to 100 c.c. with water and filtered. An excess of dry sodium carbonate is added to remove the lead. After filtration, 25 c.c. of the filtrate are mixed with 25 c.c. of water for estimating the reducing sugars by the method of Munson and Walker (*U.S. Dept. Agriculture, Bur. Chemistry, Bull.* 107 [revised], 241). Fifty c.c. of the filtrate are rendered neutral to litmus-paper by means of acetic acid, 5 c.c. strong hydrochloric acid added and allowed to stand overnight for inversion. The solution is neutralised with dry sodium carbonate and made up to 100 c.c. If necessary the solution is filtered and 50 c.c. used for the estimation of total sugars (*loc. cit.*). The amount of cuprous oxide or copper obtained in the above determinations represents the sugar (as dextrose) in 2 grms. of material. To correct for the space occupied by the insoluble material of the grain or cattle-food, the percentages of sugar found must be multiplied by 0·97, since 12 grms. of the material occupy 9 c.c. of the 300 c.c.

Sodium Carbonate Digestion Method.—The addition of sodium carbonate to the cold-water extraction method inhibits the action of enzymes on the starch, and the results obtained agree with those of the alcohol method. A mixture of 8 grm. of the finely-ground material with 190 c.c. of sodium carbonate solution (0·2 per cent.) at room temperature is taken at intervals for two and a half hours. The acidity of the material, if any, must be determined and corrected by the use of a stronger alkaline carbonate solution. Ten c.c. of neutral lead acetate solution are added, and the liquid filtered. The use of alum as a clarifier gives inaccurate results. The reducing sugars on 25 c.c. of the filtrate (= 1 grm. substance) are determined by Allihn's method (*Bull.* 107, p. 49). Fifty c.c. of the same filtrate are inverted by the acid method already described, neutralised and made up to 100 c.c., and in 25 c.c. of the solution (= 0·5 grm. substance) the total sugars by Allihn's method are determined. The amount of sucrose is obtained by multiplying the percentages of dextrose before and after inversion by the factor 1·044, then subtracting, and multiplying the difference by 0·95 as usual. No correction is usually necessary for the volume of the material, but if this be very wet, a correction may be applied for the dilution due to moisture in the sample. If only total sugar after inversion is to be estimated in substances where the amount is small, 8·8 grms. of material are extracted as above, and 50 c.c. of the solution inverted, neutralised, and 25 c.c. (=1 grm. material) used for the reduction. Reducing sugars are calculated as dextrose or as invert-sugar, and total sugars expressed as "by acid hydrolysis calculated as dextrose."

The alcohol method is very reliable, but with the sodium carbonate method care must be taken, and after clarifying the solution with lead acetate, the work must be rapidly completed as the enzymes become active again. Some materials cannot usefully be examined with this alkali method, as they do not give clear filtrates.

A. R. T.

Simple Volumetric Method for Testing Spiritus Ætheris Nitrosi. O. Herting. (*Pharm. Zeit.*, 1911, **56**, 423; through *Chem. Zentralbl.*, 1911, II., 103-104.)—The method recommended by the author for testing the strength of *spiritus ætheris nitrosi* is as follows: A mixture of 10 c.c. of water and 5 c.c. of a cold-saturated solution of potassium chlorate, free from chlorides, is placed in a glass-stoppered flask of 100 c.c. capacity; to the mixture are added 5 c.c. of the spirit to be tested, and 5 c.c. of 10 per cent. nitric acid. The whole is allowed to remain for thirty minutes with frequent shaking, and 10 c.c. of $\frac{N}{10}$ silver nitrate solution are then added. The contents of the flask are again shaken, and the excess of silver nitrate is titrated back with thiocyanate in presence of ferric ammonium sulphate. One c.c. of $\frac{N}{10}$ silver solution represents 0·0255 grm. of ethyl nitrite. J. F. B.

Valuation of Tea. A. A. Besson. (*Chem. Zeit.*, 1911, **35**, 813-815; 830-332.)—The author has analysed and examined some ninety samples of teas, of which forty-three were Chinese teas and the remainder Ceylon, Java, and Indian products. Particular attention was paid to the proportion of stalk in the samples, but it was found that there was very little relation between the amount of stalk and the price and quality of a tea as shown by a tasting test. It was ascertained, however, that Java tea grown at an altitude of 4,500 feet was of distinctly better quality than that grown at 1,800 feet. The analytical results obtained were as follows:

	Moisture.	Stalk.	Ash.	Caffeine.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Chinese green tea ...	6·00 to 7·69	0·4 to 5·3	4·88 to 7·46	2·13 to 3·22
Do. Foochow ...	6·29 to 9·06	4·1 to 17·5	4·80 to 5·73	2·23 to 3·64
Do. Hankow ...	6·48 to 8·33	8·6 to 17·1	4·95 to 5·65	2·65 to 3·64
Ceylon tea	4·57 to 8·12	5·8 to 43·4	4·54 to 5·65	2·80 to 4·10
ndian tea	4·00 to 8·08	11·5 to 37·4	4·72 to 5·64	3·31 to 4·15
ava tea	8·22 to 10·50	4·4 to 29·9	5·53 to 7·32	2·22 to 4·54

W. P. S.

Chemical Differentiation between Fermentation Vinegars and "Vinegar Essence," particularly with Respect to the Amount of Formic Acid. H. Fincke. (*Deut. Essigind.*, 1911, **15**, 145-148; through *Chem. Zentralbl.*, 1911, I., 1887.)—Most of the samples of fermentation vinegars examined by the author were found to be free from formic acid. Three samples containing pyridine also contained formic acid to the extent of 0·10 to 0·38 part per 1,000 of total acid, increasing with the proportions of pyridine. Eight samples of wine vinegar contained quantities ranging from 0·06 to 0·51 part of volatile acids capable of reducing mercuric chloride, the higher values being obtained with samples containing high proportions of wine, but whether these bodies consisted entirely of formic acid was not determined. Genuine fermentation vinegars are generally free from formic acid, but vinegars prepared from "essence" contain appreciable quantities, so that not only can fermentation vinegar be differentiated from diluted "vinegar essence" in this way, but mixtures can also be detected with some degree of certainty. Samples of "vinegar essence" showed quantities of formic acid ranging from 0·39 part to 12·04 parts—average, 4·38 parts per 1,000 of total acid. J. F. B.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of the Age of Blood-Stains. A. de Dominicis. (*Boll. Chim. Farm.*, 1911, **50**, 273-276; through *Chem. Zentralbl.*, 1911, II., 232.)—A portion of the stained material of about 5 sq. mm. is immersed in 1 c.c. of pure glycerol (sp. gr. 1·23) in a test-tube 12 mm. in diameter, which is gently given a single shake at intervals of a minute, and a note taken of the time required for solution. The yellow solution is kept protected from the air, and compared with the solutions obtained in the same way from blood-stains of known age, the relationship between the colour of which and the time required for solution is also known. The presence of blood is detected by means of the spectroscope. Blood-stains which dissolve within a minute are undoubtedly of recent origin, whilst the longer the solution period the older the stain. C. A. M.

Opsonin Reaction. H. Kronberger. (*Zeitsch. exper. Pathol. Therap.*, 1911, 9, 87-96; through *Chem. Zentralbl.*, 1911, II., 53-54.)—A method of estimating opsonins is described; the estimation is best carried out on the defibrinated blood taken from the patient and the control-subject during digestive leucocytosis. Fresh cultures of bacteria should be employed in the preparations of the emulsions, and the opsonin preparations may be stained with the ordinary stains used for bacteria and leucocytes. The reaction velocity of the opsonin is observed at intervals of ten, twenty, thirty, and sixty minutes, in a series of mixtures prepared from the bacteria and blood of the patient and the control-subject. Under these conditions the bacteria are chemotactic towards the leucocytes, and show a distinct alkaline chromatin reaction, but are negatively chemotactic towards the lymphocytes, with an acid reaction on the nucleus. W. P. S.

Biological Method for Identifying Various Fleshes. A. Saint-Sernin. (*Ann. Falsif.*, 1911, **4**, 334-338.)—The method depends upon the fact that blood-serum obtained from a rabbit which has been inoculated previously with the blood-serum of another animal possesses the property of forming a precipitate when mixed with a further quantity of the blood-serum of the animal, but not with that of any other animal. For the detection of horse-flesh, beef, or mutton, in sausages, etc., portions of the meat which are suspected to be other than pork are extracted with 0·7 per cent. sodium chloride solution for one hour, and then filtered. Portions of 2 c.c. each of the filtrate are then placed in test-tubes containing blood-serum from rabbits which have been inoculated with horse, ox, or sheep blood-serum, respectively. Should the sausages contain horse-flesh, a flocculent precipitate will form in the test-tube containing the rabbit-horse blood-serum after the tubes have been kept in an incubator for one hour at 37° C. Mutton will be indicated by a precipitate in the rabbit-sheep blood-serum tube, and so on. The active sera are obtained by injecting 10 c.c. of the necessary blood-serum into a healthy rabbit at intervals of two days, followed by three further injections at intervals of eight days. After the lapse of a week from the last injection, the rabbit is bled from the ear, and the serum separated from the blood. Certain antiseptic substances interfere with the test: acetone, formaldehyde, and sulphurous acid inhibit the reaction; thymol has less influence; whilst naphthalene, menthol, and camphor, retard slightly the formation of the precipitate.

W. P. S.

Precipitin Reaction. D. A. Welsh and H. G. Chapman. (*Zeitsch. Immunitatsforsch. exper. Therap.*, 1911, **9**, 516-529; through *Chem. Zentralbl.*, 1911, II., 38-39.)—The greater part of the precipitate obtained in this reaction consists of the constituents of the antiserum, and the weight of the precipitate is strictly proportional to the amount of the antiserum. In the case of complete precipitation the weight of the precipitate does not depend on the quantity of the other substances, and it is not correct to consider these substances (antigens) as being precipitable, or to assume that they are precipitated by the precipitin of the antiserum. Retarding effects cannot be explained by assuming that a precipitoid is formed, but at the same time heated antisera have a direct solvent action on the precipitate. The precipitin reaction is, in the author's opinion, of great practical importance, as it aids in the identification of proteins, allows closely allied proteins to be separated, etc.

W. P. S.

Use of the Quantitative "Precipitin" Reaction in Honey Analysis. J. Thöni. (*Mitt. Lebensmittelunters. u. Hyg.*, 1911, **2**, 80-123; through *Chem. Zentralbl.*, 1911, I., 1888.)—The author has investigated the method proposed by Langer, who found that von Riegler's antiserum could be employed for the quantitative examination of honey solutions. This antiserum produces a precipitate with the proteins of genuine honey, the quantity of which may be measured by volume, and thus forms the basis of a new "constant," of great utility from an analytical point of view. The formation of the precipitate for quantitative purposes is governed by certain conditions—*e.g.*, concentration of the honey solution, the relative quantities taken, and the working equivalent and quantity of the antiserum preparation. Tests carried out with ninety samples of honey and sugars showed that the sugars gave no precipitate; that artificial honeys reacted either as the sugars or else gave only traces of precipitate—*e.g.*, a sediment 2 mm. in height with 10 to 15 per cent. solutions; that genuine honeys with the same quantities of antiserum gave precipitates settling within a very narrow range of heights, and always gave a precipitate even when diluted to 1 per cent. Mixtures of genuine and artificial honey showed diminished heights of precipitate according to the extent of the adulteration. Sugar-fed honeys gave smaller precipitates than genuine pure honeys. Fermenting honeys showed no decrease in the volume of precipitate as compared with honeys in the natural condition. According to the author, the serological method is destined to prove of greater analytical value than the chemical and physical methods of examination. J. F. B.

Sterilisation of Drinking Water by Means of Ultra-Violet Rays. J. Courmont. (*Chem. Zeit.*, 1911, **35**, 806.)—It is shown that the bactericidal effect of exposure for a few seconds to the rays from a mercury vapour lamp constructed of quartz is due to the direct action of the rays, and not to the action of ozone or hydrogen peroxide. No detectable trace of ozone is formed during the few seconds' exposure required for complete sterilisation, nor even in a full minute, and the effect is shown to be independent of the presence of oxygen. The practical utility of the process is limited to ordinary drinking water, which must be free from turbidity. Whereas the rays can penetrate a foot or more of clear water and destroy all bacterial life wherever they reach, a very small proportion of any colloid causes the complete absorption of the rays by the layers of liquid nearest the lamp. Perfectly brilliant wort, beer, must, or wine, when allowed to flow over the lamp in a layer but a few millimetres in thickness, is incompletely sterilised, and it was found impossible to sterilise some broth and peptone solutions even in films less than 1 mm. thick.

G. C. J.

ORGANIC ANALYSIS.

Estimation of Amino Groups in Amino Compounds and in Urine, and a Method for the Analysis of Proteins. D. D. van Slyke. (*Ber.*, 1911, **44**, 1684-1692.)—In a previous paper (ANALYST, 1911, 17) the author described a method for the estimation of amino acids based on the measurement of the volume of nitrogen evolved in the reaction with nitrous acid. Most of the naturally occurring amino acids react quantitatively, but glycocol yields regularly 103 per cent. of the theoretical volume of gas, whilst cystine yields 107 per cent. Lysine differs from the other amino acids in the fact that it requires half an hour for complete reaction, whereas in most other cases the reaction is complete in five minutes. Among other anomalous amino compounds are included guanosine, which yields 1·25 molecules of gas, and guanine, which is so sparingly soluble that even after several hours only a portion of its amino nitrogen is evolved. With methylamine, ammonia, and the purines, about two hours are required for complete reaction. It would appear that the method under the conditions described, with a reaction period of five minutes, is only applicable quantitatively for those acids in which the amino group occupies the α-position with respect to a carboxyl group. It is not necessary to agitate the liquid during the whole time of the reaction; agitation at the end, in order to liberate the gas dissolved in the liquid, is sufficient.

In the analysis of urine, the method is applicable for the estimation of amino groups derived from proteins, peptides, hippuric acid, etc. The urea, however, reacts very slowly, complete decomposition being effected only after eight hours; but the rate of decomposition is fairly uniform, and if two experiments be made, one for six minutes and the other for twelve minutes, the difference between the two results gives a figure for the rate of decomposition of the urea which may be deducted as a correction from the first value. Normal human urine generally contains less than 1 per cent. of its total nitrogen in the form of free amino acids. The ammonia is first expelled by passing a current of air at the ordinary temperature through the liquid previously made alkaline with sodium hydroxide. The author has further developed his method for the analysis of proteins, and gives in detail the complete scheme adopted in the case of fibrin. Ten grms. of fibrin are hydrolysed with 100 c.c. of 20 per cent. hydrochloric acid, portions of 1 c.c. being withdrawn at intervals for the estimation of the amino nitrogen. When this value ceases to increase after about twenty-six hours, the hydrolysis is finished. The hydrochloric acid is then evaporated by concentration of the solution *in vacuo*, and the liquid is diluted to 250 c.c. After estimation of the total nitrogen, the various groups are dealt with separately. The amide nitrogen is estimated by distillation with milk of lime and alcohol under reduced pressure. The melanine bodies are filtered off together with the residual lime, and the nitrogen is estimated by the Kjeldahl process. The filtrate is neutralised with hydrochloric acid and concentrated. The precipitation of the hexone bases by phosphotungstic acid is carried out as previously described (*loc. cit.*), but in presence of 3·5 per cent. hydrochloric acid instead of sulphuric acid. The washed precipitate is dissolved in sodium hydroxide under certain prescribed conditions, and the phosphotungstic acid is precipitated by barium chloride, whilst the bases remain in the filtrate, which is concentrated to a volume of 50 c.c. The amino nitrogen of the bases in the solution is estimated by the author's method, allowing half an hour for the complete decomposition of the lysine. The arginine is estimated by boiling for six hours with potassium hydroxide, as described in a previous paper. After decomposition of the arginine, the remainder of the base-nitrogen is estimated by Kjeldahl's process. The sulphur contained in the cystine is estimated by the method of Benedict and Denis. In the filtrate from the bases, the total nitrogen is estimated by the Kjeldahl process, and the amino nitrogen by the author's process. If the above process for the estimation of the amino nitrogen be compared with the known processes based on the isolation of the individual amino acids, it is found in most cases that the latter processes account for barely more than half the amount of amino acids shown by the former process to be present.

J. F. B.

Reaction of Aromatic Inner Anhydrides depending on their Influence on the Crystalline Form of Iodoform. B. Bardach. (*Zeitsch. anal. Chem.*, 1911, **50**, 545-548.)—In presence of aromatic inner anhydrides and certain closely related complexes, iodoform assumes an unstable acicular form in place of the normal hexagonal plates and stars. Certain of these anhydrides give rise to this reaction direct, whilst others yield only the ordinary hexagonal crystals unless previously treated with iodine solution.

To the not too concentrated and not far from neutral solution of the substance in tap-water (the reaction only proceeds in dissociated solution), a 2 per cent. alcoholic solution of iodoform is added drop by drop. If iodoform separates in needles, aromatic inner anhydrides are present, and this reaction is especially characteristic of compounds of the type of euxanthon and its derivatives. Albumin must be removed, if present, as it acts similarly.

If hexagonal crystals are obtained under the above conditions, 3 c.c. of iodine solution (4 iodine, 6 potassium iodide, 100 water) are added to a fresh portion of the solution, and excess of ammonia (1 to 2 c.c.) added. If the ammonia completely decolorises the solution, iodine solution is added drop by drop to restore the colour. At the expiration of an hour, and after filtration if necessary, iodoform solution is added, and the solution examined microscopically. Acicular crystals of iodoform indicate the presence of aromatic inner anhydrides, such as coumarin, or of aromatic hydroxy-acids with long side-chains, or of polyhydroxyketones. G. C. J.

Estimation of Active Hydrogen in Organic Molecules. B. Oddo. (*Ber.*, 1911, **44**, 2048-2052.)—The author has introduced a gravimetric modification of Zeriwitinoff's method for the estimation of active hydrogen atoms in organic compounds by means of organo-magnesium iodides. The method depends on the reaction between ethyl-magnesium iodide and organic compounds containing OH, NH_2, NH, or SH groups, both reagents being dissolved in anhydrous solvents. For the magnesium compound the author uses isoamyl ether as the solvent, whilst for the substance to be tested anhydrous pyridine generally gives the most reliable results. In the preparation of the magnesium compound it is advisable to take rather less than the theoretical quantity of ethyl iodide, so that there may be no danger of an excess of this reagent passing away with the ethane. The apparatus employed for the estimation consists of two thin, short-necked flasks of about 50 c.c. capacity. The first flask holds the solution of the substance under examination, and is connected with the second flask by means of a tube leading from the top of the first flask to the bottom of the second. The second flask, containing the solution of the magnesium compound, is also connected with a bulb-absorption apparatus having a filter of tightly packed glass wool next to the flask, and charged with concentrated sulphuric acid. The whole apparatus is tared, together with the solutions of the reagents, and is then connected up with a U-tube containing potassium hydroxide and calcium chloride, to the further end of which is attached a bottle containing alkaline pyrogallol solution, to exclude atmospheric oxygen. A small amount of air is sucked out of the apparatus by means of the mouth, so that when the suction is relieved some of the magnesium solution passes up the tube from the second flask into the first. Reaction takes place, with evolution of ethane, and when the evolution subsides a further quantity of the magnesium reagent is passed over into the first flask. The second flask is cooled in an ice-bath, and the heat of reaction in the first flask is moderated by occasional application of cold water. Finally the reaction is completed by immersing the first flask in water at 50° to 60° C. The apparatus is cooled, disconnected from the U-tube and weighed again, the loss of weight representing the quantity of ethane evolved. This method possesses the advantage that larger quantities of the substance may be taken for the estimation than with the gasometric method. The values obtained agree sufficiently closely with the theoretical, and are calculated in terms of hydroxyl or "active hydrogen." The method is applicable in the case of oximes, camphoroxime showing 1 atom of active hydrogen. J. F. B.

Characteristic Colour Reaction of Ovolecithin. C. Casanova. (*Boll. Chim. Farm.*, 1911, **50**, 309-313; through *Chem. Zentralbl.*, 1911, II., 231.)—For the detection of lecithin the liquid under examination is heated for some time at 50° to 60° C. (to expel any alcohol which would interfere with the reaction), and extracted with ether, and the ethereal extract concentrated and treated with 2 c.c. of 10 per cent. ammonium molybdate solution, and then with concentrated sulphuric acid. In the presence of lecithin there is produced a cherry-red zone which gradually changes through greenish-yellow into deep blue. Cholesterol and phytosterol do not interfere with the reaction. In the presence of oxygen lecithin may be decomposed, through the absorption of one molecule of water into the ester of glycerophosphoric acid and choline, and by further absorption of water glycerophosphoric acid is liberated. Upon these facts is based a method of detecting adulteration, the total acidity being first estimated, and then the glycerophosphoric acid as phosphoric acid by means of uranium acetate in the ash of the ethereal extract. In an aqueous solution of lecithin the acidity rises considerably under the influence of light and oxygen, whilst in an alcoholic solution no such action takes place.

C. A. M.

Estimation of Pentosans and Methylpentosans in Cereals and Wood-Fungi. M. Ishida and B. Tollens. (*J. f. Landw.*, 1911, **59**, 59; through *Wochensch. Brau.*, 1911, **28**, 300.)—Most plant tissues contain both pentosans and methylpentosans, which yield a mixture of furfural and methylfurfural on distillation with hydrochloric acid. These bodies are estimated by precipitation as phloroglucides. But the methylpentosans yield less methylfurfural on distillation than the pentosans yield furfural; consequently, the value for methylpentosans is low if calculated by the same factor as that used for the pentosans. The methylfurfural phloroglucide is estimated by extracting it from the mixture of phloroglucides by means of hot alcohol, in which it is soluble, whereas the furfural phloroglucide is insoluble. The authors have estimated the pentosans and methylpentosans separately in a number of cereal grains and wood-fungi, and have arrived at the conclusion that if the mixed phloroglucides be calculated in terms of pentosans, as was formerly the custom, the value so obtained when multiplied by 1·1 will give the sum of the pentosans and methylpentosans present in the substance. J. F. B.

Titration of Potassium Cyanide in Presence of Potassium Ferrocyanide. W. D. Treadwell. (*Zeitsch. anorg. Chem.*, 1911, **71**, 219-225.)—Liebig's method for the titration of cyanide in presence of chloride and thiocyanate breaks down in presence of potassium ferrocyanide, which tends to dissolve silver cyanide and leads to high results. Denigès (*Ann. Chim. Phys.*, 1895, [VII.] **6**, 381) and Sharwood (*J. Amer. Chem. Soc.*, 1897, **19**, 400) have stated that addition of ammonia and a trace of potassium iodide greatly increases the sharpness of Liebig's method. The author finds the addition of the large quantities of ammonia recommended by Denigès and Sharwood are unnecessary, and even inadvisable. The conditions of success are faint alkalinity and sufficient dilution. The solution of cyanide to be titrated should not be above decinormal strength. If to such a solution, rendered faintly alkaline, 1 grm. of potassium iodide be added, the cyanide may be estimated with accuracy by titration with silver nitrate, even in presence of ten times its weight of ferrocyanide. A smaller quantity of potassium iodide (*e.g.*, 0·1 grm.) serves equally well if ferrocyanide be the only disturbing substance present, but the use of the larger quantity is recommended if thiosulphate may be present. The use of 1 grm. of iodide renders the estimation of cyanide exact in presence of half its weight of thiosulphate, but the disturbing effect of higher concentrations of thiosulphate cannot be wholly overcome by addition of iodide. The author advances a theory of the titration in support of which he adduces experimental proofs. G. C. J.

Phosphotungstic Acid Precipitation of Proteins. E. Wechsler. (*Zeitsch. physiol. Chem.*, 1911, **73**, 138-143.)—The author states that phosphotungstic acid precipitates of proteins can be readily decomposed by means of barium hydroxide if the precipitate be treated previously with acetone and water. The precipitate is completely or partially brought into solution in the mixture of acetone and water, and the barium hydroxide is then added. In some cases it is necessary to grind the precipitate in a mortar with the acetone, water, and barium hydroxide, in order to decompose it completely, whilst in other instances it is sufficient to add a small quantity of acetone to the precipitate mixed with water to obtain a readily filterable solution from which the phosphotungstic acid may be precipitated by barium hydroxide. The precipitates obtained with different proteins require various proportions of acetone and water, but a mixture of 4 volumes of acetone with 3 volumes of water will generally answer the purpose. W. P. S.

Composition of Mineral Lubricating Oils of High Boiling-Point. J. Marcusson. (*Chem. Zeit.*, 1911, **35**, 729-731; 742-744.)—The chief constituents to which mineral machine oils owe their lubricating power are those that do not react with formaldehyde and sulphuric acid. The constituents which do combine to yield "formolites" have a higher sp. gr. and refractive power, but a lower viscosity than the original oils. In applying the formolite test, 27 grms. of the oil are dissolved in 50 c.c. of normal petroleum spirit (benzine), and the solution treated, without shaking, with 30 c.c. of concentrated sulphuric acid. The mixture is cooled in ice-water, and, after the addition of 15 c.c. of 40 per cent. formaldehyde solution, it is shaken and cooled until no further evolution of heat is perceptible. The contents of the flask are allowed to stand for thirty minutes and then poured into a litre flask containing 200 c.c. of ice-water, the acid liquid neutralised with ammonia and the precipitate separated, washed with petroleum spirit until free from oil, and then with water until free from ammonia, and dried at 105° C. until constant in weight. The constituents of the oil that do not react with formaldehyde and sulphuric acid are estimated by evaporating the petroleum spirit solution and weighing the residue. They may include olefines, naphthenes, polynaphthenes and paraffins. The results thus obtained in the examination of American and Russian machine oils showed that in general the yield of formolite increased with the rise in the viscosity, in the case of refined oils. The American oils gave higher yields than the Russian oils, as is shown in the following table:

	Formolite Precipitate.	Residual Oil.	Sp. Gr. at 15° C.		$[N]_{D15°}$ C.		Viscosity at 20° C. (Engler's).	
			Original Oil.	Residual Oil.	Original Oil.	Residual Oil.	Original Oil.	Residual Oil.
	Per Cent.	Per Cent.						
American oil, refined	28·9	74·3	0·8910	0·8856	1·4962	1·4883	11·0	14·9
,, ,, ,,	30·0	75·2	0·9207	0·9064	1·5168	1·5022	24·4	28·4
,, ,, crude.	33·3	74·8	0·8994	0·8842	1·5032	1·4882	12·1	12·9
Russian oil, refined.	10·4	88·1	0·9011	0·8986	1·4950	1·4912	14·1	21·8
,, ,, ,,	17·1	—	0·9090	0·9061	1·5002	1·4962	42·5	64·2
,, ,, ,,	17·8	88·9	0·9122	0·9051	—	—	47·4	52·9
,, ,, ,,	24·4	78·7	0·9108	0·9070	1·5015	1·4968	62·0	70·8

As a rule, American machine oils contain more unsaturated hydrocarbons than Russian oils, the higher iodine value of the former being due, not only to unsaturated alicyclic hydrocarbons, but also to aliphatic hydrocarbons. On the other hand, Russian oils contain a greater proportion of saturated hydrocarbons not attacked by fuming nitric acid than do American oils. Both oils yield dinitro derivatives containing about 7 per cent. of nitrogen. The optical activity of machine oils is due, in the main, to saturated hyrocarbons, probably condensed naphthenes.

C. A. M.

Detection of Petroleum Pitch in Fat Pitch. J. Marcusson. (*Zeitsch. angew. Chem.*, 1911, **24**, 1297-1302.)—If the saponification value of a pitch-like distillation residue is lower than 33 (the minimum recorded for fat pitches), the presence of petroleum pitch or other foreign body is indicated, whilst a value of about 100 (106 highest limit for fat pitches) indicates that the product is pure. In the case of mixtures with saponification values between these limits, information is afforded by an examination of the unsaponifiable matter. Thus, if it is a powder and not viscid like the orginal material, the sample consisted of fat pitch free from petroleum residues. The same conclusion may be drawn when the oily constituents obtained after treatment with petroleum spirit and sulphuric acid do not exceed 15 per cent. of the original pitch, and have an $[N]_{D 15°}$ C. of less than 1·510. A still more reliable test may be based upon the separation of the double compound formed with mercuric bromide by the organic sulphides in petroleum pitch. Mixtures of petroleum pitch and fat pitch yield this compound, but the presence of free fatty acids retards its formation. For the detection of small quantities of petroleum pitch the following modification of the test of Malencovic is recommended: 10 grms. of the sample are dissolved in 25 c.c. of hot benzene, and the cold solution treated with 30 c.c. of $\frac{N}{2}$ alcoholic potassium hydroxide solution, and then rapidly diluted with about 200 c.c. of 96 per cent. alcohol. After standing for a short time, the solution of saponified fatty acids is decanted, and the residual pitch washed with a little alcohol, the last traces of which are afterwards removed as completely as possible with the aid of a water-pump, and by drying the residue at 105° C. The pitch is then dissolved in 100 c.c. of ether, heated beneath a reflux condenser, whilst a little calcium chloride is added to absorb any water. The solution is cooled and filtered, and the filtrate treated with 20 c.c. of mercuric bromide solution (5 grms. in 250 c.c. of ether), and allowed to stand overnight. Any deposit is collected, washed with ether, and dissolved off the filter by means of hot benzene, which will leave undissolved any mercuric bromide that may have separated at the same time. The benzene solution is evaporated, and the mercuric bromide double compound will be left as a dark brown or black spongy mass, which when heated in a tube decomposes with the formation of a spongy mass of carbon, and in which sulphur may be detected by the usual method (distinction from mercuric bromide.) Coal-tar pitch forms a similar compound, but may be distinguished from petroleum pitch by methods previously described (see ANALYST, 1911, 227), whilst brown coal-tar pitch may be recognised by Graefe's diazobenzene chloride reaction (ANALYST, 1906, **31**, 204; 1908, **32**, 477; 1911, *loc. cit.*). C. A. M.

Estimation of Rosin in the Residues from the Distillation of Coal Tar. Holde and F. Meister. (*Chem. Zeit.*, 1911, **35**, 793-794.)—From 10 to 20 grms. of the mixture of coal-tar distillation residue and rosin (or 2·5 to 5 grms. when the amount of rosin exceeds 4 per cent.) are extracted with 200 c.c. of ether beneath a reflux condenser, and the insoluble residue washed three times with ether. The filtrate is shaken with successive portions of 30 c.c. of $\frac{N}{10}$ sodium hydroxide solution until the aqueous layer appears colourless, the resulting soap solution shaken twice with 50 c.c. of ether, and the united ethereal extracts once more shaken with 30 c.c. of the alkali solution. The united alkaline extracts containing the colophony are shaken with ether and an excess of dilute sulphuric acid in a separating funnel, the ethereal solution of the liberated abietic acid is washed free from mineral acid, after the addition of concentrated sodium sulphate solution, then filtered, concentrated to 100 c.c. on the water-bath, heated for about ten minutes with 0·5 grm. of dry bone charcoal to remove the dark resinous substances derived from the coal tar, and filtered. The filtrate and washings are evaporated, and the residue dried for five minutes at 105° C. and weighed. An addition of 8 per cent. is made for the substances in the colophony not soluble in the sodium hydroxide solution. This correction has been empirically determined. The results thus obtained with test mixtures containing from 1·5 to 14 per cent. of rosin were in close agreement with the theoretical amounts. C. A. M.

Detection of Salicylic Acid. H. C. Sherman and A. Gross. (*J. Ind. and Eng. Chem.*, 1911, **3**, 492-493.)—The ferric chloride reaction for salicylic acid is not characteristic of this substance, the compounds maltol and iso-maltol, present in small quantity in certain foods, giving a similar coloration. Jorissen's reaction has been found useful for distinguishing these substances, the authors' modified method of carrying out the test in the case of small quantities of salicylic acid being as follows: To the solution to be examined 4 or 5 drops of sodium or potassium nitrite solution (10 per cent.), the same quantity of acetic acid (50 per cent.), and 1 drop of copper sulphate solution (1 per cent.) are added, shaking after addition of each reagent. The solution is heated for forty-five minutes in a boiling-water bath, the liquid in the tube being completely immersed in the hot water. After allowing to cool, the tube is examined against a white background both vertically and horizontally, and is compared with a blank test with water to which the reagents have been added. By this test 0·005 to 0·010 mgrm. of salicylic acid can be detected. If considerable amounts of salicylic acid are present, a stronger solution of copper sulphate may be used, and for qualitative results a shorter time of heating is sufficient. The red colour of this test is stable, and the results are quantitative. The authors confirm Allen's observation that benzoic, cinnamic, and tartaric acid do not give a coloration, and find that maltol, iso-maltol, methyl-ethyl-aceto-acetate, orcin, arbutin, resorcin, and phlorizin, all of which give some coloration with ferric chloride, do not respond to this test. A. R. T.

Analysis of Shellac and Shellac Varnish. A. C. Langmuir and F. S. White. (*J. Soc. Chem. Ind.*, 1911, **30**, 786-789.)—The authors return to the question as to the comparative merits of the Hübl and Wijs methods of determining the percentage of iodine absorbed by mixtures of shellac and rosin. Both methods are shown to be affected by time and temperature of the reaction. The authors adhere to previously published figures as to the iodine values for these substances, and hold that the Wijs method is more accurate, since there is a difference of 210 units between its values for shellac and rosin (228 and 18), while in the case of the Hübl method this difference is only 115 (125 and 10). (*Cf.* ANALYST, 1905, **30**, 164). The Hanus modification possesses no advantage over these methods. Endemann's condensation method (ANALYST, 1907, **32**, 425) for the determination of the purity of shellac is stated to be unreliable.

The authors determine the proportion of *wax* in shellac as follows: 100 grms. of the sample are dissolved in 500 c.c. of water containing 20 grms. of sodium carbonate. The liquid is decanted from insoluble matters, and the suspended wax taken out of the liquid by adding a known weight (about 15 grms.) of paraffin wax to the boiling liquid. After standing till cold, the paraffin cake is removed, dried, and weighed. Small detached particles of paraffin wax are recovered by filtering the liquid, drying the residue, washing the wax through the filter by means of petroleum spirit, and evaporating the solvent, when the residue is weighed and the weight added to the main portion. The increase in weight of the paraffin gives the amount of shellac wax.

Arsenic may be determined by the two following methods, according as it is present in large or small amounts:

1. Two grms. of shellac are boiled with 50 c.c. of strong sulphuric acid and

15 grms. of potassium sulphate until all organic matter is destroyed, when the solution is diluted and hydrogen sulphide passed through the liquid. The precipitated arsenic sulphide is filtered, dissolved in ammonium carbonate, and the solution acidified, when the pure sulphide is dried and weighed. Some arsenic is dissolved by fused shellac, and the above method gives the total arsenic. Orpiment may be estimated by dissolving 100 grms. of shellac in alcohol, and washing by decantation. The residue is then oxidised by nitric acid, and the solution evaporated with sulphuric acid till fumes are evolved. Hydrochloric acid and ferrous sulphate are then added and the liquid distilled as usual, and the arsenic determined in the distillate.

2. The detection of traces of arsenic may be made by a modification of the Gutzeit or Marsh-Berzelius method. Ten grms. of the shellac are dissolved by heating with 50 c.c. of arsenic-free nitric acid, and, after a few minutes' boiling, 15 c.c. of arsenic-free sulphuric acid are added, and the liquid boiled till fuming. A practically dry mass of carbon is left. This is evaporated with 50 c.c. of saturated sulphurous acid solution to dryness, and 60 c.c. of water are then added, the liquid boiled down to 40 c.c., and the solution filtered and made up to a known bulk (75 c.c.). An amount of solution corresponding to 2 grms. of shellac is taken, and the arsenic determined by colorimetric comparison in the Gutzeit or similar apparatus.

Careful sampling from a large bulk of shellac is shown to be necessary, different portions frequently containing widely different proportions of rosin. The sp. gr. of an orange shellac was found on immersion to be 1·1141, and by floating 1·1350, at 15·5°/15·5° C. The difference is due to the presence of numerous "air-cells" in shellac.

Shellac Varnish.—Wood spirit and denatured grain alcohol are the usual solvents. The boiling-point of varnish prepared with the former, determined by heating the varnish in a large test-tube and holding the thermometer just above the liquid, will approximate 68° C., and 78° C. for denatured grain spirit.

To detect methyl alcohol in grain alcohol, the presence of acetone from the wood alcohol added to the grain alcohol is relied on. The alcohol is distilled from 150 c.c. of sample, and then redistilled, using a Linnemann fractionating tube, collecting the first 5 c.c. This is diluted to 100 c.c. with water, and 4 c.c. of the solution heated in a boiling water-bath for ten minutes with 4 c.c. of a solution of 5 grms. of precipitated mercuric oxide in 100 c.c. water and 20 c.c. sulphuric acid. A white precipitate, appearing in a few minutes, is produced in the presence of acetone. One per cent. of denatured alcohol in the grain alcohol may be detected, and 2 per cent. gives a distinct reaction.

The weight or body of shellac varnish is expressed in pounds of shellac per gallon of alcohol taken. It may be determined by weighing 0·5 to 1 grm. in a weighing-bottle, transferring with alcohol to a weighed dish containing 10 grms. ignited sea sand and a stirring-rod, and heating for just one hour at 110° C. Since even at this temperature shellac holds some water, the weight of residue is multiplied by 0·96 in the case of white shellac, and 0·98 for orange shellac.

The addition of water or "benzine" to the alcohol in shellac varnish may be determined by taking the sp. gr. of the distillate, or by diluting the varnish, when the "benzine" will separate. Spirit-soluble copals, sandarac, and rosin are added to shellac varnish as adulterants. Wijs's iodine solution, diluted with 2 parts by volume of glacial acetic acid, may be used to detect these substances. A few drops of varnish are added to 2 to 3 c.c. of this diluted Wijs's solution. Pure shellac remains unchanged in colour, except on long standing. Rosin gives an immediate reddish coloration. Copal and sandarac yield a red colour (less pronounced than in case of rosin) and a curdy precipitate. This precipitate is also formed when an alcoholic solution of copal or sandarac is diluted with glacial acetic acid, or when a large excess of alcohol is added. Shellac wax settles after dilution, but it can be distinguished from the above precipitate. The odour of copal and sandarac, when the varnish is rubbed between the fingers, is characteristic. If rosin alone is present with the shellac, a rough approximation of its amount, by comparison with standards, may be made with the above colour test. An alcohol-soluble casein preparation, met with as an adulterant, was detected by means of the high proportion of ash, which contained boric acid. A. R. T.

INORGANIC ANALYSIS.

Estimation of Arsenic and Iron Salts in Mineral Waters. F. Ageno and N. Guicciardini.—(*Gazz. Chim. Ital.*, 1911, **41**, 473-478.)—The author describes an indirect method of estimating arsenic and iron salts in mineral waters by means of acid permanganate. C. A. M.

Detection and Estimation of Calcium Cyanamide in the Presence of Other Fertilisers. L. Vauflart. (*Ann. Falsif.*, 1911, **4**, 321-324.)—Calcium cyanamide may be detected in mixtures containing organic and other substances by the odour of acetylene which is evolved when the mixture is moistened (calcium cyanamide usually contains traces of calcium carbide), by the black carbonaceous residue remaining after extraction with water, and by the yellow precipitate which is formed when an aqueous solution of the fertiliser is treated with silver nitrate; this precipitate is insoluble in ammonia, soluble in nitric acid, and frequently contains a small quantity of silver acetylide, which remains as a black powder when the yellow precipitate is dissolved in nitric acid. Treatment of the fertiliser with mineral acids also affords information of the presence of calcium cyanamide and of the character of some of the other constituents. As regards the estimation of cyanamide, the author finds that, whilst Brioux's method (ANALYST, 1910, **35**, 486) yields satisfactory results in the case of cyanamide itself (on an average, 87·3 per cent. of the total nitrogen is found to be present as cyanamide nitrogen), the results obtained in the case of mixtures are much too low. This is due to the fact that the rate at which cyanamide is converted into dicyandiamide is considerably accelerated in the presence of such substances as superphosphate, etc. If, however, both the cyanamide nitrogen and the dicyandiamide nitrogen be estimated as described by Brioux (*loc. cit.*), the result obtained will be a measure of the calcium cyanamide in a mixture, it being taken into account that only 87·3 per cent. of the cyanamide nitrogen is thus found. For instance, a fertiliser consisting of phosphate, potassium salts, calcium cyanamide, and dried blood, and stated to contain equal quantities of the two latter substances, yielded 5 per cent. of total nitrogen. The cyanamide (*plus* dicyandiamide) nitrogen amounted to 2·3 per cent., corresponding with 17·7 per cent. of calcium cyanamide. As 17·7 per cent. of calcium cyanamide containing 15 per cent. of total nitrogen would yield 2·65 per cent. of nitrogen, the difference between the latter figure and the total nitrogen represents the organic nitrogen; this amount, 2·35 per cent., corresponds with 19·5 per cent. of dried blood containing 12 per cent. of nitrogen (*cf.* ANALYST, 1911, 429). W. P. S.

Oxidation of Ferrous Salts. C. Baskerville and R. Stevenson. (*J. Amer. Chem. Soc.*, 1911, **33**, 1104-1106.)—Experiments are described which show that the tendency of ferrous salts in acid or neutral solution to undergo oxidation by the air has been greatly exaggerated. In one experiment a stream of air was passed for three hours through a solution containing 0·7 grm. of ferrous iron, and at the end of that time only 0·1 mgrm. of ferric iron was found. Addition of chromium, copper, manganese, nickel, titanium, vanadium, and tungsten, and of the ordinary dust of the laboratory air, had no measurable accelerating influence on the oxidation. The conclusion is drawn that ferrous iron, in acid and neutral solution, at room temperature, is so slowly oxidised, and that this oxidation is so slightly affected by such substances as are apt to be present, that expulsion of air after reduction by zinc is unnecessary, and the titration may be performed at leisure in ordinary determinations of iron by means of permanganate. G. C. J.

Volumetric Estimation of Lead Peroxide. J. F. Sacher. (*Chem. Zeit.*, 1911, **35**, 731.)—The method of Lux (*Zeitsch. anal. Chem.*, **19**, 153), in which red lead is decomposed with nitric acid, the liquid boiled with oxalic acid to dissolve the lead peroxide, and the excess of oxalic acid titrated with permanganate, gives too low results, owing to the fact that a portion of the lead peroxide is dissolved by the nitric acid. To the same cause must also be attributed the errors in the electrolytic estimation of lead as peroxide from a nitric acid solution, whereas by adding oxalic acid prior to the nitric acid the method is satisfactory. The lead may also be precipitated by means of sulphuric acid before the titration with potassium permanganate, as suggested by Tocher (*Farben-Zeit.*, 1899-1900, 214). There is no need to separate the lead sulphate, and the analysis may be completed in twenty to twenty-five minutes. Forestier's gravimetric method of estimation (*Zeitsch. angew. Chem.*, 1898, **11**, 176), which is based upon a treatment of the red lead with hot acetic acid and estimating the insoluble residue of lead peroxide either gravimetrically or volumetrically, gives inaccurate results, since lead peroxide is reduced by hot acetic acid. C. A. M.

Estimation of Nitrate. G. Romijn. (*Zeitsch. anal. Chem.*, 1911, **50**, 566-570.)—Dilute solutions of nitrates are reduced quantitatively to nitrite by pure zinc dust in presence of ammonia and an ammonium salt. The nitrite is then determined volumetrically, or, if present in minute amount only, colorimetrically. Iron salts must be absent, or partial reduction to ammonia may result. Into a 100 c.c.

measuring flask there is placed 5 c.c. of a solution made by dissolving 100 grms. of ammonium sulphate in water, adding 100 c.c. of ammonia (sp. gr. 0·96), and diluting to 300 c.c. The flask is next filled to the mark with the water to be tested, which should not contain more than 400 mgrms. NO_3 per litre. A mixture in equal proportions of pure zinc dust and kieselguhr (200 mgrms.) is added, the whole well shaken, and allowed to stand five minutes. The solution is filtered, the first runnings rejected, and 50 c.c. of the clear filtrate taken for the determination of nitrite. If this be large in amount, 10 c.c. of $\frac{N}{10}$ permanganate and 5 c.c. of sulphuric acid (sp. gr. 1·124) are shaken with the solution in a stoppered flask, which is then set aside for fifteen minutes. Potassium iodide (0·5 grm.) is then added, and the liberated iodine, equivalent to the excess of permanganate taken, is estimated by means of thiosulphate. Smaller amounts of nitrite are determined colorimetrically, and for this purpose the author recommends the use of a dry mixture of α-naphthylamine (1 part), sulphanilic acid (10 parts), tartaric acid (100 parts). This mixture keeps indefinitely, and, when using it, no other acid need be added. G. C. J.

Rapid Method for the Estimation of Nitrates. A. Quartaroli. (*Staz. sperim. agrar. Ital.*, 1911, **44**, 157-164; through *Chem. Zentralbl.*, 1911, II., 49-50.)—The method depends on the action of anhydrous formic acid on nitrates, according to the equation:

$$2KNO_3 + 6H.COOH = N_2O + 4CO_2 + 5H_2O + 2H.COOK.$$

From 0·1 to 0·3 grm. of the nitrate is gently heated with 5 c.c. of formic acid in a test-tube of 15 c.c. capacity; this tube is provided with a cork and delivery-tube, and the evolved gases are collected over mercury in a nitrometer. After the volume of the gas has been noted, 2 c.c. of concentrated potassium hydroxide solution are introduced into the nitrometer, the latter is shaken, and the volume of the gas is again noted. The first reading gives the volume of the nitrous oxide and carbon dioxide, the second that of the nitrous oxide alone. The temperature and pressure being known, the quantity of nitrate in the sample may then be calculated. The results obtained agree closely with those found by the Schulze-Tiemann method.

W. P. S.

Action of Hydrazine Sulphate on Nitrites, and a Method for the Estimation of Nitrite Nitrogen. B. B. Dey and H. K. Sen. (*Zeitsch. anorg. Chem.*, 1911, **71**, 236-242.)—When excess of hydrazine sulphate is allowed to act on a solution of any ionised nitrite, nitrogen, nitrous oxide, and ammonia result, in accordance with the following equation:

$$2NH_2.NH_2 + 3ON.OH = NH_3 + 2N_2O + N_2 + 4H_2O.$$

If the reaction be allowed to take place in a nitrometer, the ammonia and a considerable proportion of the nitrous oxide (depending on the amount of water present) remain in solution. If the gas be washed three or four times with 20 c.c. of water, the last of the nitrous oxide may be removed and the residual nitrogen measured. This nitrogen is two-thirds of that originally present as nitrite. The method has been applied successfully to some fifty nitrites including tetramethylammonium nitrite, benzylamine nitrite, as well as the nitrites of the alkalis, alkaline earths, and heavy metals. On the other hand, amyl nitrite, and, in general, non-ionised nitrites, do not give rise to any evolution of gas when brought in contact with hydrazine sulphate. G. C. J.

Analytical Examination of Commercial Oxygen. C. Baskerville and R. Stevenson. (*J. Ind. and Eng. Chem.*, 1911, **3**, 471-476.)—The compressed oxygen from a cylinder was first passed through a trap to catch any solids and liquids, and in this trap were contained red and blue litmus-paper and starch-potassium-iodide paper, for the detection of acids, alkalis, halogens, and ozone. Moisture was determined quantitatively by means of two weighed U-tubes next in order in the series, these tubes containing phosphorus pentoxide on glass-wool, and the second one being a check on the accuracy of the first tube. A calcium chloride tube was then interposed to protect these moisture-tubes from the moistening-bottle. This bottle, next in order, contained a moist, porous, cotton rope, through which the gas is passed, the rope being at its lower end immersed in the water in the bottle. The moisture in the rope is insufficient to absorb impurities from the gas, but it allows of a repeat test on *moistened* gas with the previously-mentioned reaction papers, which in the form of three successive cones were placed in the tube leading from the moistening-bottle. The gas was next passed through nearly-saturated caustic baryta solution, which showed the presence or absence of carbon dioxide, and at the conclusion of the test was divided into three parts for the following tests: (1) SO_3, after addition of hydrochloric acid; (2) for halogens and halogen acids and oxides of chlorine, by addition of nitric acid and silver nitrate; (3) this was set aside. Caustic soda solution (2:3) was in the next container, and after the experiment this was tested as follows, after division into three parts: (1) for CO_2, SO_2, and SO_3, by addition of barium chloride, and hydrochloric acid in excess; (2) for SO_2, H_2S, halogens, halogen acids, and chlorine oxides, by acidification with nitric acid, addition of ammonia till alkaline, then silver nitrate, and reacidification with nitric acid; (3) this was mixed with the reserved portion of the baryta solution, and tested for hydrocyanic acid and cyanogen by the Prussian blue test.

The gas was next passed through silver nitrate solution, which repeats some of the previous tests, and also shows PH_3, SbH_3, and AsH_3. Next two calcium chloride tubes were used to dry the gas, which was then passed through the CuO furnace, thus oxidising the hydrogen and any organic compounds. Two weighed calcium chloride tubes take up the water formed, and the carbon dioxide was obtained by weighing the next apparatus containing baryta solution. Solid caustic soda in the next tube served as a guard for the baryta apparatus. The gasometer received the gas after passing through the various items of the apparatus, and the oxygen was there measured, 10 to 18 litres passing in three to six hours.

A separate experiment was made for the following: A trap containing litmus-paper and starch-KI paper repeated the previous observations. Next the gas passed through starch-KI solution as a test for ozone, and portions of this solution were tested before and after with methyl-orange and phenolphthaleïn to indicate acids and bases in the gas. A slightly acid solution of ferrous sulphate was next in order, and this was titrated before and after the passage of the oxygen to determine active oxidising compounds, oxygen having no effect under these conditions. Very dilute permanganate solution, lastly, served to detect reducing agents. Oxygen itself was determined direct by alkaline pyrogallol solution, and the nitrogen and rare atmospheric gases obtained by difference. N_2O could not be tested for. Organic matter was generally absent. Carbon dioxide was separately determined by absorption in sodium hydroxide solution (2 : 3) saturated with oxygen. Analyses of seven samples of commercial oxygen gave results showing 92·8 to 99·2 per cent. of oxygen, 0·25 to 6·9 per cent. of nitrogen and inert gases, moisture up to 0·15 per cent., CO_2 up to 0·11, hydrogen 0·14 per cent. (only in one sample), and all other impurities absent.

A. R. T.

Detection of Perborates and Similar Compounds. W. Lenz and E. Richter. (*Zeitsch. anal. Chem.*, 1911, **50**, 537-544.)—To distinguish sodium perborate from borax, a little potassium bichromate solution is added to the aqueous solution, which is then shaken with ether. In presence of perborate the ether is coloured blue by perchromic acid. The reaction is due to hydrogen peroxide, and is given by potassium percarbonate, but not by ammonium persulphate, potassium perchlorate, or periodic acid.

Solutions of perborates (which are alkaline, owing to partial decomposition by water) decolorise permanganate with separation of manganese peroxide. In presence of acid no separation of peroxide occurs, but the colour of the permanganate is discharged. Percarbonates (and hydrogen peroxide) behave similarly, and perborates and percarbonates can be titrated with permanganate. Ammonium persulphate in acid solution has no action on permanganate in the cold, and the action is feeble at higher temperatures; in alkaline solution it slowly decomposes permanganate in the cold, with separation of manganese peroxide, and the action is increased by heating. Potassium perchlorate in acid solution has no action on permanganate; in alkaline solution it slowly transforms the violet colour to blue, the action being more marked at higher temperatures. Periodic acid has no action on permanganate.

When silver nitrate in not too small amount is added to a solution of sodium perborate, a heavy, black precipitate separates at once. This is easily washed by decantation, and is then insoluble in 10 per cent. ammonia. Solutions of perborates acidified with nitric acid give no reaction with silver nitrate. In ammoniacal solution, silver nitrate gives no immediate precipitate, but on standing, and more quickly on warming, gas is evolved, a silver mirror deposited, and the solution acquires the blue colour of molecular silver. Potassium percarbonate gives a white precipitate with silver nitrate in the cold, but the colour passes through yellow to grey on warming. The precipitate is insoluble in 10 per cent. ammonia. In nitric acid solution, percarbonates give no reaction with silver nitrate, nor do they give any reaction in an ammoniacal solution until this is boiled, when the blue colour of molecular silver appears. Ammonium persulphate gives this blue colour in neutral solutions, whilst in nitric acid solution it is coloured yellow, and in ammoniacal solution silver nitrate decomposes it with evolution of gas, but without the formation of any precipitate. Potassium perchlorate is unacted on by silver nitrate. In aqueous solution, periodic acid gives with silver nitrate a yellow precipitate, consisting of doubly refractive rhombohedra, which are soluble in ammonia, but not in a cold solution of thiosulphate and sulphurous acid. In cold ammoniacal solution, periodic acid gives, with silver nitrate, a faint brown coloration, which gives place to a dark brown precipitate on warming.

The capacity to peroxidise manganous hydroxide was determined as follows: Into each of a number of test-tubes, 3 to 5 c.c. of a 1 per cent. solution of manganous sulphate was placed, dissolved oxygen expelled by boiling, the solution cooled to 60° C., covered with petrol, and allowed to cool completely. A few drops of caustic soda solution were then added to the contents of each tube, when a nearly white precipitate resulted. To one tube 3 to 5 c.c. of ordinary distilled water, not freed from air, was added as control, and to the others 3 to 5 c.c. of each of the solutions to be tested. Sodium perborate and percarbonate in 0·08 per cent. solution caused a distinct darkening of the precipitate; potassium persulphate and periodic acid in 0·02 per cent. solution produced an equal darkening, whilst potassium perchlorate had no effect.

Cobalt hydroxide, similarly protected from aerial oxidation, was tried as reagent, 3 to 5 c.c. of a 0·25 per cent. solution of the nitrate being precipitated by caustic soda. In a blank experiment the precipitate passes from blue to pale pink, and darkens very slowly. Two drops of a 1 per cent. solution of sodium perborate produce a distinct brown colour, and the same tint is given by 6 drops of potassium percarbonate or 1 drop of ammonium persulphate (all in 1 per cent. solution). Potassium perchlorate and periodic acid are without effect.

A 1 per cent. solution of nickel sulphate, treated in a similar manner to the cobalt nitrate, gave no reaction with perborate, percarbonate, perchlorate, or periodic acid. On the other hand, it proved a very sensitive reagent for ammonium persulphate, as little as 2 drops of a 0·1 per cent. solution giving rise to a black precipitate.

Sodium perborate and potassium percarbonate and perchlorate give no reaction with basic lead acetate solution, whereas periodic acid gives a heavy, white, microcrystalline precipitate, even in very dilute solution. Ammonium persulphate in concentrated solution gives after a time a yellow precipitate with basic lead acetate. Hydrogen peroxide renders basic lead acetate solution turbid and dark, but the reaction is not given by dilute solutions, and disappears on boiling.

Solutions of cerous chloride, to which a few drops of ammonia have been added, are coloured yellow by hydrogen peroxide, sodium perborate, and potassium percarbonate. The reaction will detect 0·03 mgrm. hydrogen peroxide, 0·5 mgrm. perborate, or 2 mgrms. percarbonate. Ammonium persulphate, potassium perchlorate, and periodic acid, give no reaction.

In absence of free acid, only ammonium persulphate of the salts here considered liberates iodine from potassium iodide. In presence of sulphuric acid, perborate and percarbonate act similarly, but perchlorate is without effect. None of the salts liberate bromine from potassium bromide.

Sodium perborate and potassium percarbonate decolorise a solution of 0·05 grm. fuchsin and 12 grms. sodium acetate in 100 c.c. water. The colour is not restored on dilution, but the decolorised solution acquires a yellow tint when acidified with acetic acid. Ammonium persulphate slowly decolorises fuchsin, but to observe the reaction the fuchsin solution must be added drop by drop to a large bulk of the persulphate solution. Potassium perchlorate is without action on fuchsin.

When a 1 per cent. solution of sulphuric acid is saturated with aniline, filtered, mixed with an equal bulk of a 1 per cent. solution of perborate or percarbonate, and heated to boiling, a faint yellow colour is developed. Ammonium persulphate in 1 per cent. solution gives rise to a dark brown when tested in a similar manner, and as little as 0·5 mgrm. may be detected by means of aniline sulphate. The reagent is unaffected by perchlorate. G. C. J.

Volumetric Estimation of Phosphoric Acid. J. Rosin. (*J. Amer. Chem. Soc.*, 1911, **33**, 1099-1104.)—Of the methods depending on the precipitation of phosphoric acid by silver nitrate, with subsequent estimation of the excess of silver by Volhard's method, that of Holleman (*Zeitsch. anal. Chem.* 1895, **34**, 85) is the best; but the results are too high owing to co-precipitation of silver acetate if a large excess of sodium acetate is used, and too low if only a small excess of sodium acetate is used. In contradiction to the statements in the textbooks, the reaction between silver nitrate and sodium phosphate is shown to proceed in accordance with the equation: $3Na_2HPO_4 + 6AgNO_3 = 2Ag_3PO_4 + 6NaNO_3 + H_3PO_4$. This phosphoric acid must be neutralised if its precipitation by silver is to be complete, and zinc oxide serves well for the purpose, but the use of a great excess must be avoided. The solution in which phosphoric acid is to be estimated is neutralised (to phenolphthaleïn) by means of caustic soda free from chloride or of known chloride content, and an excess of $\frac{N}{10}$ silver nitrate is added, and the mixture well shaken in a 200 c.c. measuring flask. The amount of silver nitrate added should be at least 30 per cent. in excess of that required, but the addition of two or three times the theoretical amount introduces no error. Zinc oxide, or a suspension of it in water, is next added little by little, shaking vigorously for two or three minutes after each addition, and testing for free acid each time by dropping in a fragment of blue litmus-paper. When the colour of the litmus-paper is no longer affected, the solution is made up with water to the mark, well mixed, and allowed to subside for a few minutes only, as zinc oxide acts slowly on silver nitrate, precipitating it as oxide. The mixture is filtered, and the excess of silver determined in 100 c.c. of the filtrate by Volhard's method. The extreme error of the method is ± 0·1 c.c. of $\frac{N}{10}$ solution. The degree of dilution within the limits 100 to 250 c.c. has no measurable influence on the results.

The phosphoric acid in precipitated calcium phosphate may be determined with an accuracy of 1 in 500 by shaking for a few minutes with at least twice the theoretical amount of $\frac{N}{10}$ silver nitrate, adding zinc oxide, again shaking, making up to known volume, filtering and determining the excess of silver in an aliquot portion of the filtrate. Addition of zinc oxide is unnecessary if the sample consists wholly of tricalcic phosphate, but this is rarely the case. Chloride, if present, must be determined separately, and a correction made. Nitrates and sulphates do not interfere, but the method cannot be used for the determination of total phosphoric acid in presence of more than traces of iron and aluminium. G. C. J.

Titration of Phosphates. W. Strecker and P. Schiffer. (*Zeitsch. anal. Chem.*, 1911, **50**, 495-499.)—The authors have investigated two methods for the volumetric estimation of phosphoric acid in phosphates, namely, titration with uranium acetate and with silver nitrate. A solution containing a known amount of pure sodium phosphate was employed in the experiments, and the results obtained were compared with those yielded by the gravimetric molybdate method; the quantities of phosphoric acid found by the three methods in equal volumes of the phosphate solution were: Molybdate method, 0·0605 grm.; uranium method, 0·0604 grm.; silver nitrate method, 0·0604 grm. These results show that the volumetric methods are not less accurate than the gravimetric method. W. P. S.

Rapid Determination of Silver and Cadmium by Means of the Gauze Cathode and Stationary Anode. R. C. Benner and W. H. Ross. (*J. Amer. Chem. Soc.*, 1911, **33**, 1106-1112.)—The use of the gauze cathode with stationary anode, which was found to be so efficient in the determination of nickel and cobalt (ANALYST, 1911, 307), has been extended to include silver and cadmium. The range of electrolytes from which these elements could be deposited on the gauze in adherent form, when strong currents were used, was found to be much less than in the case of nickel and cobalt. The deposits obtained for each metal with potassium cyanide as electrolyte were entirely satisfactory.

For the deposition of silver, the electrolyte should contain about 7 grms. of potassium cyanide and 2 grms. of potassium hydroxide in 50 c.c. The potassium hydroxide prevents the appearance of a red colour due to the decomposition of cyanide, and in the amount named has but little effect on the rate of deposition. If it be desired to deposit more than 0·3 grm. of silver, more cyanide must be employed to insure an adherent deposit, but increasing the amount of cyanide decreases the rate of precipitation to a marked extent. Under the conditions named, with a current of 5 ampères, 0·15 grm. silver may be deposited in ten minutes. Before breaking the current the electrolyte is replaced by water, otherwise an error of − 0·4 mgrm. may result. The extreme error of the method is ±0·5 mgrm.

For the deposition of cadmium the electrolyte may contain 2 to 5 grms. of potassium cyanide and 1 to 2 grms. potassium hydroxide and 0·1 to 0·3 grm. cadmium in each 60 c.c.; or Stoddard's directions (*J. Amer. Chem. Soc.*, 1909, **31**, 385) may be followed and potassium cyanide added in amount just sufficient to dissolve the precipitate first formed, potassium hydroxide being next added until the solution is distinctly alkaline. When working according to Stoddard's directions the deposition of 0·2 grm. of cadmium is complete in twenty-five minutes using a current of 4 ampères. When the larger quantities of potassium cyanide and hydroxide are used, it is necessary to wash before breaking the current. The extreme error of the method is ±0·6 mgrm. G. C. J.

Colorimetric Method for Vanadium in Iron and Steel. C. R. M'Cabe. (*Chem. Engineer*, 1911, **13**, 243-247.)—In absence of chromium or tungsten 2 grms. of the steel are dissolved in 40 c.c. of hot strong hydrochloric acid, 5 c.c. of nitric acid

(1 : 1) are added, and the mixture evaporated to about 10 c.c. and transferred to a separator, the dish or beaker being washed with hydrochloric acid (1 : 1) until the liquid in the separator amounts to 20 c.c. Ether (50 c.c.) is added, and the mixture shaken and allowed to separate. The lower layer and 1·5 c.c. of the ethereal layer are then drawn off and diluted with 200 c.c. of boiling water, after which ammonia is added. The bulk of the iron remains in the ethereal layer in the separator, but drawing off 1·5 c.c. of this layer with the aqueous layer insures the presence of about 0·1 grm. of iron, which is desired. When addition of ammonia has precipitated the iron, a further 10 c.c. of ammonia are added and boiling continued for half a minute. The whole of the vanadium and iron is precipitated, whilst manganese remains almost wholly in the solution, when this is heated to boiling before addition of ammonia. The iron and vanadium precipitate is filtered off and, if nickel is present, is redissolved in hot dilute hydrochloric acid and reprecipitated. In absence of nickel the filter containing the precipitate is opened in a dish and treated with 3 c.c. hydrochloric acid (1 : 1) and 7 c.c. water, warming moderately and rocking until solution is complete. The contents of the dish are transferred to a stoppered glass tube, until with washings this contains about 40 c.c., and then 0·5 c.c. of hydrofluoric acid, previously diluted with water, is added, when the ferric solution loses its colour. Hydrogen peroxide (5 c.c. of a solution containing 0·5 per cent. H_2O_2) is next added, and the solution diluted until its brown colour is exactly equal to that of a solution of vanadic acid of known strength. Directions for the preparation of vanadic acid, which is not obtainable commercially, are given. The standard solution contains 0·2 mgrm. vanadium in each c.c., and in preparing dilute solutions for colour comparisons a measured quantity of a dilute ferric solution containing about 0·1 grm. iron is acidified with 3 c.c. hydrochloric acid (1 : 1), decolorised by hydrofluoric acid, a measured quantity of the standard vanadium solution (usually 15 c.c. at first) is added, and the whole diluted to five times the measure of the vanadium solution taken.

In presence of chromium the acid solution, after ether separation of most of the iron, is evaporated almost to dryness, 50 c.c. of nitric acid are added to the residue, and the solution boiled until fumes are all expelled and the solution is a clear green. To the boiling nitric acid solution 2 or 3 grms. potassium chlorate are added to precipitate manganese and oxidise chromium. Boiling is continued for a few seconds, after which 200 c.c. of boiling water are added, and the iron and vanadium precipitated by means of ammonia. The iron and vanadium are separated from manganese, nickel and a trace of chromic acid by resolution in hot, dilute hydrochloric acid and reprecipitation by means of ammonia in dilute boiling solution. A trace of chromium usually remains with the iron and vanadium, but does not interfere.

In presence of tungsten the filtrate from the tungsten determination is used for the estimation of vanadium. Since the evaporation to dryness in the tungsten determination leads to partial reduction of vanadium, it is necessary to re-oxidise this by adding 5 c.c. nitric acid (1 : 1) during evaporation of the filtrate preceding the ether separation of iron. Sulphuric acid must not be used in the tungsten determination.

Titanium and molybdenum do not interfere with the above method.

G. C. J.

Simultaneous Volumetric Estimation of Iron and Vanadium. E. Müller and O. Diefenthäler. (*Zeitsch. anorg. Chem.*, 1911, **71**, 243-249.)—Campagne (*Ber.*, 1903, **36**, 3164) described a method for the estimation of vanadium, which consisted in reducing V_2O_5 to V_2O_4 by repeated evaporation with hydrochloric acid, finally evaporating to fuming with sulphuric acid, after which the solution was diluted and titrated with permanganate at 60° C. The authors obtained results which were from 5 to 10 per cent. low when following Campagne's directions, and conclude that he omitted some detail of importance from his description. The following method gives results which are accurate within less than 1 drop of $\frac{N}{10}$ permanganate: To the solution of vanadate, contained in an Erlenmeyer flask, 15 c.c. of hydrochloric acid and 50 c.c. of alcohol (or 15 to 20 c.c. of an alcoholic solution of hydrogen chloride) are added, and the mixture boiled until of a pure blue colour. It is then transferred to a basin and evaporated to dryness on the water-bath, and the residue heated for a further ten to fifteen minutes on the water-bath, with frequent trituration with a rod. This treatment eliminates organic matters which, on treatment with sulphuric acid, would yield substances having a reducing action on permanganate. The dry residue is covered with 5 c.c. sulphuric acid, which is then heated until it fumes. The solution is then allowed to cool, diluted and titrated with permanganate.

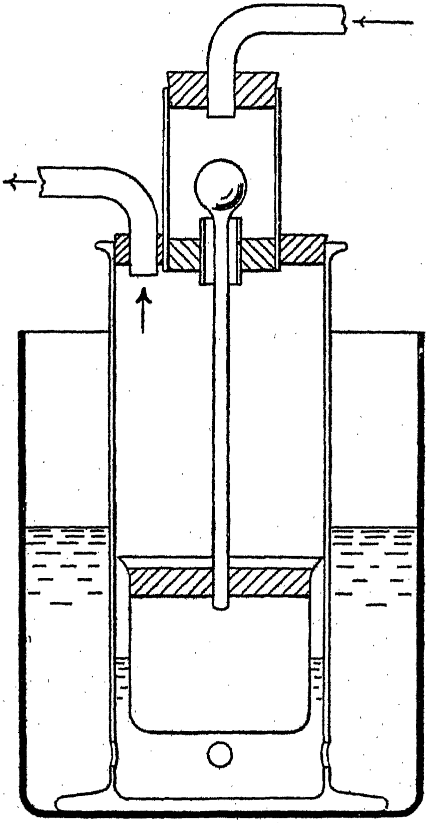
Applied to ferro-vanadium, the above method leads to high results, as in presence of much solid matter it is impossible to eliminate the whole of the organic matter after the evaporation. As this organic matter has no measurable reducing action itself on permanganate, but only gives rise to reducing substances on heating with sulphuric acid, the use of sulphuric acid should be avoided in presence of iron, and may with advantage be avoided in every case if Reinhardt's solution (phosphoric acid and a manganese salt) is available. The alloy (1 grm.) is dissolved in a little nitric acid, the excess of which is partially evaporated and the last traces of it destroyed by addition of hydrochloric acid and further evaporation. Hydrochloric acid and alcohol are then added, and the mixture boiled in a flask until the vanadic acid is all reduced, and finally evaporated in a basin to 5 c.c., not to dryness. The solution is made up to known volume, and, in an aliquot portion, vanadium is estimated by titration with permanganate in presence of Reinhardt's manganese mixture. In another portion of the solution iron and vanadium may be estimated together, after reduction by means of sulphurous acid; or the iron may be estimated somewhat less accurately, but more conveniently, by an iodometric method with which the reduced vanadium solution does not interfere. G. C. J.

Determination of Vanadium in Vanadium and Chrome-Vanadium Steels. J. R. Cain. (*J. Ind. Eng. Chem.*, 1911, **3**, 476-481.)—As the result of disagreement between the results obtained on the same sample by the staff of the United States Bureau of Standards and several other chemists, an investigation of the methods most in use for the estimation of vanadium was undertaken by the Bureau, and the results communicated to the other chemists, with whose co-operation the true vanadium content of the sample was soon agreed on, and certain sources of error in the determination of vanadium traced. These sources of error are set out fully in the present paper, in which it is stated that methods requiring the use of ferrocyanide as indicator for ferrous salts, where quadrivalent vanadium and ferric salts are also present, must of necessity be very uncertain. An error in all methods where final titration is made against permanganate may arise by failure to deduct the blank caused by the presence of other elements than vanadium. This error may amount to 100 per cent. in the case of low-vanadium products. The temperature of titration, which should be as near 80° C. as can be arranged without risk of exceeding that temperature, is also important if a sharp end-point is to be secured. In methods in which iron is removed by ether extraction, ferrous salts are apt to be formed, and must be re-oxidised. If the evaporation of a solution of quadrivalent vanadium with strong sulphuric acid to eliminate hydrogen chloride, be too prolonged or conducted at too high a temperature, oxidation may occur. In methods in which, after an ether separation, the remainder of the iron, as well as any chromium, etc., is to be separated from vanadium by pouring into a boiling solution of caustic soda, there are several sources of possible error. Vanadium is almost always carried down with the hydroxides of iron and chromium, and two or three precipitations are necessary. Another source of error is due to manganese, which tends to become oxidised to peroxide during the boiling with soda, and this peroxide oxidises chromium salts to chromate, the separation of which is so difficult that the following new method is recommended:

The borings (2 to 4 grms.) are dissolved in 40 to 60 c.c. of dilute (1 : 10) sulphuric acid in a covered Erlenmeyer flask. The insoluble matter, which may contain most of the vanadium, is fused for a few minutes with bisulphate, and the aqueous solution of the fusion added to the main solution. The solution is nearly neutralised with sodium carbonate (saturated solution), and then finely-divided cadmium carbonate is added in small portions, at intervals of four or five minutes, to the solution, which is kept boiling and covered. One or 2 grms. of cadmium carbonate should remain undissolved. About fifteen to twenty minutes' boiling suffices. The precipitate, which contains all the vanadium and chromium, very little iron, and no manganese, is allowed to settle, filtered rapidly, and washed (twice only) with hot water. It is then dissolved in the minimum quantity of nearly boiling dilute sulphuric acid (1 : 10), catching the filtrate in the original flask. The cooled solution is nearly neutralised with ammonia, leaving only so much free acid as will prevent any iron from precipitating on boiling. Cadmium is next removed by heating the solution to boiling and passing hydrogen sulphide, and, after allowing to settle, filtering. The precipitate is washed two or three times with hot water, and the filtrate and washings concentrated to 60 to 70 c.c. and electrolysed, using a mercury cathode and a current of 5 to 6 ampères at 6 to 7 volts. The solution is tested for iron by ferricyanide, and absence of iron is usually a safe indication that all chromium as well as iron has passed into the mercury. With much chromium present relatively to iron, 3 to 4 c.c. of the electrolyte may be withdrawn, a few drops of hydrogen peroxide added, and the solution boiled for a few minutes after the brown colour, due to vanadium peroxide, has disappeared. If the solution remains clear and colourless on adding ammonia, the electrolysis is ended. The test portion is acidified with sulphuric acid, and added to the main solution. The exhausted electrolyte is withdrawn, and the mercury washed two or three times with 25 to 30 c.c. of water while the current is passing. To the united solutions 2 to 3 c.c. of sulphuric acid (1 : 10) is added, the mixture heated to 70° to 80° C., and permanganate added until a strong pink colour results. The vanadium is reduced by leading sulphur dioxide into the boiling solution, the excess of sulphur dioxide expelled by continuing to boil whilst passing carbon dioxide, and the solution is then filtered through asbestos, cooled to 70° to 80° C., and titrated with $\frac{N}{50}$ permanganate. With special facilities a determination may be completed in one and a half hours. G. C. J.

APPARATUS, ETC.

Simple Forms of Gas-Pressure Regulators. E. Stansfield. (*Chem. News*, 1911, **104**, 3-4.)—The author describes two patterns of regulator. In each there is (1) an outer vessel; (2) an inner cylinder open top and bottom, and which may consist (see Fig.) of a cylindrical vessel with one or more holes drilled through the sides near the bottom, or of a wide-mouthed glass-bottle with the bottom cut off; a piece of wide glass-tube could be used; (3) a float, consisting of a beaker, and weighted at the base so that it floats upright: the rim of the beaker should be an easy fit inside the cylinder; (4) gas inlet and outlet tubes; (5) a valve connected to the float: this valve consists, in the apparatus figured, of a small bulb blown at one end of a glass tube, the other end of which is fixed in a cork in the neck of the float. The valve seating is a short bit of glass tube. If the end of this tube is first made square by rubbing on emery-cloth, a little grinding of the valve on its seating will make it nearly airtight.

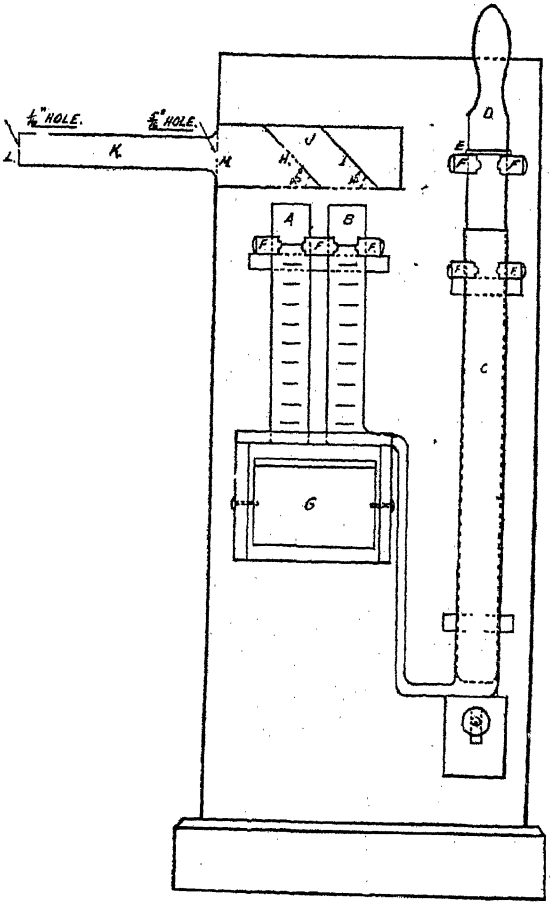


Water is poured into the outer vessel and gas passed through in the direction indicated by the arrows. More water poured into the outer vessel will give a higher pressure, and *vice versa*.

The other regulator has inlet and outlet gas tubes blown into a vertical tube. The valve is a glass rod fixed in the cork of the float at one end, and having a cylindrical cap at the other, which fits in the vertical tube closely, yet allowing free movement. A piece of glass tube with a cork in it, in which the rod is fixed, makes a suitable cap. The inner vessel and accessories of the regulators should be fixed in a cork fitted in the neck of the outer vessel, to prevent upsetting. The area of cross-section of the float should be large as compared with the valve, so that the pressure exerted on the top of the valve by the inlet gas is negligible, or a large excess of inlet over outlet pressure might permanently close the valve. The type shown in the figure gives a more constant pressure than the second form of apparatus.

A. R. T.

New Colorimeter. E. D. Campbell and W. B. Hurley. (*J. Amer. Chem. Soc.* 1911, **33**, 1112-1115.)—The unknown solution is placed in the tube A, which is 19 cm. long, 3 cm. in diameter, and graduated for 15 cm. The standard solution is placed in the tube B, which has the same dimensions as A, the graduated portion being divided into 100 divisions of 1·5 mm. each. In one piece with B are a connecting tube and a reservoir, C, in which the glass plunger D works, so that the level of the liquid in B can be controlled. The plunger has a rubber collar, E, to prevent it from descending too far and breaking the bottom of the reservoir. A, B, and C rest on wooden supports, that under A and B being provided with holes for the passage of light reflected from the adjustable mirror G. F, F are spring clips for holding the glass parts in place whilst allowing of their easy removal for cleansing. The black wooden back of the colorimeter serves the purpose of a support for the parts of the instrument, and of a screen as it is interposed between the colour tubes and the source of light (a north window). The light passing through the tubes impinges on the mirrors H and I cemented to brass plates sliding in grooves cut at an angle of 45° in the sides of the wooden box, J, which is supplied with a loose cover. The mirror H is cut vertically and cemented in such a position as to reflect one half of the circular field of light coming through A. The light passing up through B is reflected horizontally by the mirror I through a hole in the brass plate supporting H. One half of the circular field of light from B is cut off by the mirror H, the vertical



edge of which acts as a dividing line between the two halves of the circular field. The image of one half of the tube B is then observed in juxtaposition to the opposite half of the image of the tube A. The juxtaposed images are observed through a tube K, 2·5 cm. in diameter and 16 cm. long, lined with black felt and provided with an eyepiece having a hole 1·5 mm. in diameter. At the point M in the tube K is placed a diaphragm having an aperture 8 mm. in diameter. All parts inside the box J, except the mirrors, are painted black. By having the apertures in the eyepiece and diaphragm properly proportioned, only the image of the bottoms of the tubes A and B can be seen, thus preventing interference of light reflected from the vertical sides of A and B. An observer looking through the eyepiece sees a single field divided vertically by an almost imperceptible line when the two solutions are of the same intensity. By manipulating the plunger D, the level of the liquid in B can be raised or lowered, thus causing the right half of the image to assume a darker or lighter shade at will. G. C. J.

Laboratory Apparatus for the Production of a Constant Current of Ozonised Air. L. v. Liebermann. (*Chem. Zeit.*, 1911, **35**, 734.)—A stout test-tube is fixed by means of a perforated cork into one of the limbs of a U-tube which has side tubulures. This test-tube is closed by a stopper through which passes a platinum wire terminating in a piece of platinum foil. The other limb of the U-tube is closed by means of a cork, and the whole is placed in a strong glass vessel (battery jar) containing sulphuric acid of about 1·4 to 1·5 sp. gr. This acid is also introduced into the test-tube to the same level. An asbestos cover, with openings to admit the top of the U-tube, is placed over the battery vessel, and through another opening in this is passed a platinum wire also ending in platinum foil. Both wires are connected with a battery, whilst a current of dry air is drawn through the side tubulures of the U-tube. It is advisable to saturate all the cork connections with paraffin wax to prevent their being attacked by the ozone. C. A. M.

GOVERNMENT REPORT.

Report to the Local Government Board on the Nutritive Value of Bread made from Different Varieties of Flour. J. M. Hamill. (*Food Reports*, No. 14. Cd. 5831. Price 3d. 1911.)

The Report deals solely with "genuine" flours obtained by mechanical means from the wheat grain without chemical treatment or the addition of foreign substances (*vide* Food Report, No. 12, Cd. 5613, by Hamill and Monier-Williams, ANALYST, 1911, 254).

I. *Classes of Wheat Flour.*—A short account of the morphology of the wheat grain (illustrated) is followed by a discussion of milling methods and the quality and grading of flour. The whole of the complicated machinery of modern milling is devised with a view to obtaining in white flour nothing but the contents of the parenchymatous cells of the endosperm, although absolute separation is never effected in practice. Products other than flour are termed *offal.* *Bran* consists of the pericarp and aleurone layer with some adhering parenchymatous cells of the endosperm; *pollard* is bran in a finer state of division; *fluff, or fibre,* consists largely of the cellulose walls of the endosperm cells; *sharps, or middlings,* are mixtures of pollard, fluff, and germ fragments. By modern methods 70 to 73 per cent. of ordinary white flour can be obtained from an average cleaned wheat; bran and pollards account for about 20 per cent., and finer offal, sharps, and germ for 10 per cent. The extent to which all these are separated from each other depends on their market value. The old process of stone milling is only suitable for the softer or mellower wheats with tough skins, as, owing to the severe attrition to which the berries are subjected, the brittle skins of hard foreign wheats become too much disintegrated for effective subsequent separation. Flours refined to different degrees may be obtained from stone-milled wheat, but the amount of separation ordinarily obtained by this method contrasts unfavourably with that reached in roller-milling, in which the grain is reduced to flour in gradual stages, and the offal is continually removed by sifting and the use of air-currents. More efficient separation of bran and a larger yield of high grade flour thus becomes possible. Contrary to common notions, high grade flour is subjected to less grinding and coarser dressing than the lower grades, which are dressed through very fine silk. A variety of different grades of flour can be made to suit any demand by dividing the output of the roller mill in different ways as it is being produced; thus it is perfectly easy to prepare so-called "standard flour" from such mills by suitable adjustments. Much uncertainty exists on the subject of "germ" amongst millers. In stone-milling it appears certain that much of the germ passes away into the offal, only the more finely-ground particles passing through the coarse mesh used for dressing such flour. Roller-milled flour is practically free from germ. Millers generally are averse to returning the germ to flour, alleging that its presence has a detrimental effect on the baking and keeping qualities of the flour and on the stability and strength of the dough, and that it is impossible to produce a large, well-risen loaf from flour containing germ. In addition, it is declared to be unsuitable for use with aërating substances such as baking-powder, owing to the development of unpleasant flavours, and that flour containing it soon acquires a rancid flavour. The question of keeping quality cannot, however, be regarded as definitely settled, the superiority in this respect of modern flour being probably due to improved methods of milling and the use of sounder wheats. There is evidence to show that flours to which the germ is returned keep well provided they do not contain an excessive amount of moisture, and such flours, when containing germ in amounts insufficient to affect the colour of the bread, acquire a pleasant, nutty flavour. Many fancy flours contain large amounts of added germ, thereby enhancing their protein content; the germ in such cases is previously subjected to heat in order to destroy its enzymic activity and to prevent any development of rancidity. The highest grades of roller-milled flour absorb more water and produce a larger loaf with a better colour and appearance when judged by the usual criteria applied by the baker and the public than the lower grades of roller-milled flour, and, judged by the same criteria, both are superior to stone-milled flour.

As regards flavour, bread from stone-milled flour is often considered superior to that made from roller-milled. The presence of more germ in the former, and the fact that English wheat which has a good flavour is that usually employed, probably accounts for this. Subtle enzymic changes occur, due to the presence of germ, favoured by warmth and keeping. In breads made from either stone or roller milled flour, loss of flavour, as well as liability to become harsh and dry, are frequently ascribed to "over-proving"—*i.e.*, over-fermentation—in an attempt to obtain as large a loaf as possible. The greater the percentage yield of flour obtained from wheat, the darker will be the colour of the loaf, due to the presence of offal; other factors influencing colour are the mill employed (stone or roller) and the colour of the wheat used—red or white. It follows that different flours containing the same percentages (over 70 per cent.) of the total wheat may yield loaves varying much in colour. The great bulk of the flour used in this country is the product of roller mills, although stone-milled flour is still made, and there is a distinct, though limited, demand for it.

The classes of milling products from which bread may be made are as follows: (1) *Wholemeal, or "Graham," flour* is produced by grinding the entire wheat grain. Obviously, it should contain the whole of the germ and bran particles. No sieves or bolting-cloths are used in making it. (2) *"Entire" wheat flour, or fine meal,* is a product obtained by removing a portion of the bran, and finely grinding the rest of the grain. It can also be prepared from stone-rolled wholemeal by sifting. The amount of offal removed in either case varies. In the United States, flour produced from wheat with a portion of the husk removed is known as "entire" wheat flour, and this inaccurate term is used in the report, as the greater part of the experimental work quoted has been done in that country. (3) *Households* grade of flour is the commercially lower grade of flour obtained from roller mills. It is darkish in colour, contains a little finely-divided bran, and, from the baker's point of view, is inferior to high grade flour for bread-making. Flour of this type is known as *baker's grade* and *clear grade* flour in America. (4) *Patent grade* flour is the commercially higher grade flour produced by roller mills. The water-absorbing capacity of such flour, and the size and general appearance of loaves made from it, appeal strongly to bakers. Whereas a certain grade of flour may yield ninety loaves per 280 pounds of flour, one hundred loaves may be obtained from higher grades as a result of the absorption of more water. (5) *Straight-run, or patent grade,* flour is the whole of the flour produced in the roller mill. It is intermediate in appearance and baking qualities between households and patent grades. (6) *Special flours, generally sold under fancy names,* may contain added germ (usually cooked), finely ground bran, lentil flour, banana meal, etc.

II. *Nutritive Values of Various Milling Products (Meals and Flours) and Bread made therefrom.*—Numerous tables, mostly from the publications of the United States Department of Agriculture, are given, showing the chemical composition and heats of combustion of various samples of wheat, flour, and bread. A flour relatively rich in protein may, as a result of absorbing more water, yield a bread containing less protein than bread derived from flour relatively poor in protein. High grade flours have a high water-absorbing capacity. The caloric value of bread is approximately five-eighths of the caloric value of the flour from which it is made. Germ consists of about one-third protein, one-third carbohydrate, and one-sixth oil, ash and water accounting for the residue. Since wheat does not contain more than 2 per cent. of actual germ, the inclusion of the whole of it would only raise the apparent protein content of the flour about 0·6 per cent., and this last figure would fall to 0·2 or 0·3 per cent. if only such germ were returned to the flour as it is possible to separate from the branny offal. The protein in the germ contains no gliadin or glutenin, but a considerable amount of nucleic acid in combination with albumin and globulin, and the organically combined phosphorus in the nucleic acid is mainly confined to the germ. The combinations in which such elements as sulphur, phosphorus, and iron, are found in the ash gives no indication of the form in which they were present in the flour. The ash contents of bread are very variable relatively to the ash contents of the flours from which they were made, generally being about half of the mineral matter present in the original flour. It is not possible to base conclusions as to the nutritive value of different breads merely upon chemical data, physiological experiments being necessary to determine the digestibility of the different materials. It would appear that patent grade flour is more easily acted upon by the digestive juices *in vitro* than "entire," or Graham flour. Numerous tables are given, comparing the digestibility of different kinds of flours and breads, as actually determined in feeding experiments on individuals. The principal conclusion arrived at is that the differences in nutritive value between various grades of flour made from the same wheat are usually insignificant when compared with the differences in nutritive value of flours made from wheats differing widely in composition.

The mineral requirements of the body are next discussed, as are the recent experiments at Cambridge and Liverpool, where white and wholemeal breads were fed to rats and pigeons. In these two cases the animals failed to thrive on white bread and flour, but did better on wholemeal. The bearing of these experiments and those of Leonard Hill (*Brit. Med. J.*, 1911, 1310) on the connection known now to exist between polished rice and the disease of beri-beri (H. Schaumann, *Beihefte zum Archiv f. Schiffs- und Tropenhygiene*, 14; Beiheft 8) is pointed out. No conclusive evidence is available as to the relative merits of wholemeal and white flour on the teeth.

III. The question of so-called "standard flour" is touched upon, and the definition of it given—80 per cent. of the wheat, with all the germ and semolina—adversely criticised. The various methods in which it is compounded are described, and the difficulty shown of giving any definition of such material apart from a preliminary "standard wheat." Many so-called "standard flours" consist of mixtures of low grade flour with fine offal bought for the purpose, and their use results in a profit of from 2s. 6d. to 3s. per sack, as compared with 80 per cent. straight-run flour. An endeavour to obtain uniformity by prescription of physico-chemical limits, sufficiently stringent to be of value, would inevitably rule out a considerable portion of the wheat at present imported. It would seem that "standard" flour (regarded as 80 per cent. straight-run together with the germ) has advantages over the "imitation standard" in the palatability and appearance of the loaf made from it. Tables are given containing the results of analyses by G. W. Monier-Williams, which show the chemical composition of seven distinct wheats and of the various milling products as well as "standard" flour obtained from them. The differences in apparent protein and mineral matter between the "standard" and household flours obtained from the same wheat are comparatively small in all cases.

Extracts from the Conclusions.—"The records of experimental work which have been consulted for the purposes of this report, and other writings on the subject, do not permit any conclusive statements to be made regarding the exact value in nutrition of different varieties of flour."

"The notion that ordinary high grade and naturally white 'patent' flour is practically devoid of protein or nitrogenous constituents, whereas the latter are abundantly present in bread made from wholemeal and 'entire' wheat flours, is erroneous. The differences which exist in this respect are not relatively of great magnitude, and they may to a large extent be neutralised by imperfect absorption from the digestive tract. A 'patent' flour derived from one variety of wheat may contain considerably more total protein, and furnish more available 'energy than an entire' wheat or wholemeal flour from another kind of wheat. There is no reason to believe that the varities of bread which the miller and baker have accustomed us to regard as of lower quality—'households,' for example—are in any physiological sense inferior to that of the higher priced bread made from the high grade and specially white flour. On the contrary, from the point of view of available nutrient material and energy value, the advantage is on the side of the 'households.'"

"'Entire' wheat flours (including stone-ground flours and 'standard' flour) are in nearly the same position as 'households,' although when made from weak wheats they will usually contain less available protein than 'households' made from strong wheats. They possess, however, additional constituents, due to the presence of branny particles and the germ of the wheat, which appear to have a value of their own in nutrition, and may, as a result of further investigation, be shown to comprise phosphorus-containing organic compounds or other substances, the presence of which in some part of the dietary, even in minute quantity, is important in maintaining good health."

"For children who live largely on bread, or bread supplemented by jam, sugar, or other foods, which add little to the available mineral matter, proteins, organically-combined phosphorus, or other substances which possibly may be necessary for health, there appears on the balance to be advantage in the use of bread made from flour of the 'entire' wheat class, or from wholemeal in which the bran is very finely ground. It should, however, be remembered that many children whose food consists largely of bread do not get enough of it, and are really underfed in respect of such essential nutritive substances as proteins and carbohydrates. To increase the quantity of bread taken in such cases may be of greater importance than the substitution of one form of bread for another; to supplement the bread by other articles will be still more useful." H. F. E. H.

* * * * *

REVIEWS.

A SYSTEMATIC HANDBOOK OF VOLUMETRIC ANALYSIS. By FRANCIS SUTTON. Tenth Edition, revised by W. LINCOLNE SUTTON and ALFRED E. JOHNSON. London: J. and A. Churchill, 1911. Pp. xiv + 621. Price 21s. net.

It may seem a work of supererogation to review the tenth edition of a standard work like that of Sutton, but as the author states in his preface that at the age of fourscore years he is not equal to the task of revision, and has placed it in the hands of his son and Mr. Johnson, it is to some extent a new work.

When this book was first published, nearly fifty years ago, analytical chemistry had hardly attained the status of a scientific profession; the analyst differed from the pure chemist in his training, in his methods, and largely in his mode of thought. But as time went on the training of the analytical chemist became more and more scientific; great strides were made in both branches of the profession, especially during the interval that has elapsed since the publication of the ninth edition, and to-day the analyst fairly ranks as a scientific chemist. In 1863, Sutton was rather in advance of his times, and for many editions he kept his work up to date, and progressed with the times. He expresses himself in the preface particularly anxious that the tenth edition "should be distinguished by the most thorough and critical revision, and, in the result, by the greatest possible consonance with modern practice," and the revisers had a splendid opportunity to rewrite the book in the light of the advances of the past few years. It cannot be said that they have fully availed themselves of this opportunity.

The old conception of an indicator was that it gave an end point to a reaction; the laws of mass action teach us that it is really an equilibrium point, which will approximate more or less to the completion of the reaction according to the conditions. This idea should form the basis of volumetric analyses, and might have been simply explained quite early in the book in the section of alkalimetry by describing the modern theories of indicators; the revisers, however, simply refer to the publications of Ostwald and Noyes, and do not attempt even an abstract of their work. Had they done so, the explanation of very many methods would have been greatly simplified, such as the titration of ferrous salts with permanganate in the presence of chlorides, and the principles underlying Gay-Lussac's method for the assay of commercial silver would have followed as a matter of course, instead of being quite wrongly stated.

The nomenclature is most confused: thus, methyl-orange is described as sodium dimethylamido-azo-benzene sulphonate, and methyl-red as *p*-dimethylamino-azobenzene *o*-carboxylic acid; in various parts of the book the prefix "para" is variously denoted *p*-, p, **p**, and para; and α-β-γ- propane triol is referred to indiscriminately as "glycerol," "glycerin," and "glycerine"; such expressions as "sulphocyanate," and "dihydric potassium phosphate" occur. There is often a looseness of expression, such as "Sulphuric acid, in a dilute form, has no prejudicial effect on the pure permanganate."

There are many other annoying features of the book: the working details of the methods, which are the portions of the book that will be most often consulted, are printed in so small a type as to be a little difficult to read; the index is very incomplete and inconveniently arranged; references to figures in the text are often without the page on which they are found; and references to original papers are most unsystematic—thus, on p. 45 one finds J. S. C. I, **12.** 432, and J. S. C. I, 1899. 708; while, as a variation, on p. 48 the reference is J. S. C. I. 1900. **19.** 958, the last obviously being the best form.

There is a remarkable blemish in the section on Gas Analysis, where ethane and hydride of ethyl, as well as butane and hydride of butyl (propane presumably not existing, but only hydride of propyl) are referred to in more than one place as distinct substances of different series, C_nH_{2n+2} and $(C_nH_{2n+1})_2$, and there is a serious misprint on p. 525, which may possibly lead to accidents, it being stated that "the hydrocarbons $(C_nH_{2n+1})_2$ and C_nH_{2n+2} may be absorbed by absolute alcohol, some of which is introduced into the absorption tube, and *ignited* for a short time with the gas"; probably *agitated* is meant. Much modern work—for instance, that of Bone and his colleagues, and of Butterfield—is ignored, and no mention is made of benzene in illuminating gas.

In some cases recent methods have been overlooked—for instance, in the section on Indigo, the tetrasulphonate method of Bloxam is not given; in others, obsolete methods are described at too great a length, such as Pavy's method for the estimation of reducing sugars; and it is a debatable point whether methods like that of Polenske for the estimation of insoluble volatile acids in butter and other fats, the final stage of which happens to be volumetric, would not be better left to special textbooks.

Notwithstanding what has been said, there is very much that is good in this book. Methods, nearly all of them good, are described for the estimation of almost every substance, and no analyst, working by the directions given, which are clear and sufficient in detail without being prolix, will be misled. With this excellent ground-work, it should not be a difficult task to make the next and Jubilee edition—to which the author is looking forward, and which all chemists hope he will be spared to see published—worthy of its great reputation. If modern chemical ideas, such as the law of mass action and the ionic theory, are borne in mind; if recent work on gas analysis is studied; if a more systematic system of nomenclature, as that of the Chemical Society, is adopted; if the working details are printed in larger type, leaving the smaller for the explanations of the methods; and if the services of a competent indexer can be secured, the work will realise the ideals of its venerable author.

H. DROOP RICHMOND.

AN INTRODUCTION TO THE CHEMISTRY OF COLLOIDS. By Dr. VICTOR POESCHL. Translated by HERBERT H. HODGSON, M.A., B.Sc., Ph.D. London: Chas. Griffin and Co., 1910. Price 3s. 6d. net.

This is a translation of a small textbook, which in the original has reached a second edition, and may be useful in giving readers unfamiliar with the large works of W. O. Ostwald and of Freundlich a general view of the subject, the importance of which is certainly growing.

The first two chapters are devoted to the general characteristics, nomenclature, and properties, of colloidal solutions. These are described on lines generally received, except for a somewhat daring attempt to explain the Brownian movement (pp. 10 and 11), which takes no notice of the universally accepted results of Einstein and of Perrin, but is certainly not an improvement. Then follow two further theoretical chapters on the relations of colloidal to true solutions and to suspensions, and on classification, W. Ostwald's division into suspensoids and emulsoids—*i.e.*, systems consisting of one liquid and one solid phase and of two liquid phases respectively—being used. These theoretical considerations are now interrupted by two chapters devoted to the preparation of colloidal solutions and to research methods—viz., chiefly to ultra-microscopy. The first-named gives a brief but fairly complete survey of the various substances so far obtained in colloidal solution, as well as the methods employed in their preparation. The chapter on ultra-microscopy will, perhaps unavoidably, be almost unintelligible to readers not fairly familiar with the instrument itself. Theoretical considerations are resumed under the heading "Recent Views on the Nature of the Colloidal State," a great part of which must make very difficult and not very useful reading to students without previous knowledge of the subject. Two brief concluding chapters deal with the importance of colloids in various sciences and industries, a somewhat disproportionate amount of space being devoted to mineralogy.

As will appear from the foregoing, the arrangement of the matter is not particularly systematic, and, in addition, the language is occasionally so lax that the reader is confused or misled. As far as p. 32—*i.e.*, through about one-third of the book—the term "colloid" is used indifferently, either in Graham's sense as denoting a *form*, and in the modern as denoting a *state*, of matter. Only then the latter meaning is definitely made clear. In several places (pp. 12, 31, etc.) "fluorescence" is described as a fairly general property of colloidal solution, and is tersely explained as "colours diffusing [*sic*] by transmitted from reflected light." Even after due allowance for the obvious misprint, this is certainly an excessively careless statement.

As regards the translation, it is a glaring specimen of a too common type, in which the guiding principle is to retain all the eccentricities of German construction, while translating every word by the first English equivalent given in the dictionary. The result is a complete absence of idiom, style, and, occasionally, of sense. No reader unfamiliar with German can possibly guess what phenomenon is meant by "the scattering of colour in the ultramicroscopic image." "Through the methods worked out by R. Zsigmondy and H. Siedentopf . . . we have been able to orientate more exactly the cause of heterogeneity"; "Berthelot observed that a hundred billionths of a gramme of iodoform could still be scented"; "On heating the gels part with their adsorption ability," are a few further specimens of the method used in translating.

Until, however, some of the larger standard works are available in English translations, the book may be useful, notwithstanding some original and acquired defects, in stimulating the interest of readers unfamiliar with German.

E. HATSCHEK.

OILS: THEIR COMPOSITION, MANUFACTURE, AND ANALY
MMONS, B.Sc. (London), and C. AINSWORTH MITCHELL, B.A. (
London: Scott, Greenwood and Son. 1911. Price 7s. 6d. net.

authors say in the preface: "The aim of the present volume is to des a concise manner the properties of different edible fats and oils and their c ations, and to give an outline of the modern processes used in their preparati d purification." It is obvious that such an extensive subject can only be ver briefly outlined in the limited space of some 150 pages, part of which ha not been utilised to the very best advantage, owing to certain matters having been dealt with twice under different sections. The first chapter is clearly expressed, and contains well-selected formulæ and tables; in Chapter II. eighteen fats and oils are described in eight pages, with a very limited number of analytical "constants" and "variables," and unfortunately (with three exceptions) only one set of figures is given in each case, so that the reader does not receive the very necessary information as to how far a genuine sample may depart from these examples. In some cases, however, a few more figures are given for such of these oils as are again dealt with throughout the work, where other fats are also described.

We are told that Arachis oil is only "occasionally" used in margarine, and it is hard to understand the extraordinary statement—in several places throughout the book—to the effect that cocoa butter (or other fat) is largely used for "the *cream* of chocolate creams."

Under refining, bleaching, and deodorising, an excellent outline is given of most of the more important manufacturing methods, and it is correctly suggested that other unpublished processes are used, the secrecy of which is jealously guarded. The chapters on butter and lard are perhaps the best in the book, and contain much useful information. The authors very properly point out that "the discoveries of science . . . have led to the practice of a considerable amount of very skilful adulteration," and they express the hope "that the somewhat lengthy chapter on the analysis o od products will be useful to those engaged in the industry, in enabling such adulter to be detected and successfully combated." In the chapter in question, v analytical methods are given in a more or less detailed manner, and most of t important recent methods are described, but it is regrettable that in some value to the analyst is greatly diminished by misprints, mistakes, and omiss little practical details which are absolutely essential to one who wants to the methods from the instructions given. A few of these may be cited—the Ze tyro-refractometer number of butter-fat is given as 35° to 38° at 40° C.; t onification *value* of cocoa butter as 286 to 290, where saponification *equival* ntended. Under the Polenske process (notwithstanding the vast amount of w lished in this country and in Germany, indicating the great importance of b grade and quantity of powdered pumice, as well as the author's own warning he necessity for strictly specified conditions) there is but little excuse for uctions to add "a few fragments of pumice." There is a misprint of 100 d of 110 c.c. in the amount to be distilled in the given time. In the Kirsc s, where 10 c.c. of dilute sulphuric acid and 35 c.c. of water should be ad f dilute sulphuric acid is erroneously given and no mention is made of

Two methods for estimating boron preservative are de
would break down for boric acid, seeing that no alkali is ad
eas the more recent method of Richmond and Miller (ANALYST,
mentioned.

Under chocolate, it seems somewhat outside the scope of the work to inc
r. Booth's proposed standards and to describe even in such a sketchy and insuffici
anner methods for the estimation of various non-fatty constituents.

On considering the work generally, and more particularly from the analytical standpoint, one cannot help feeling that much of the useful data it contains is somewhat depreciated in value by the want of a little more revision and attention to detail in putting them together, while in a few cases there is a suggestion of a want of personal verification by the authors. Nevertheless, the work is full of useful information, with abstracts from a very wide expanse of scientific literature, and interesting statistics are given in the last chapter.

E. R. BOLTON.

THE PRINCIPAL STARCHES USED AS FOOD. Second Edition. Illustrated by Photomicrography. By WALDRON GRIFFITHS. Cirencester: Bailey and Woods. 1911. Price 6s.

This little book consists of some twenty-five photo-micrographs of the principal starches printed on the Kodak Company's "Vigorous Art" Velox Paper. The results may be described as very satisfactory, excellent definition having been obtained in all but one or two instances, and even in the case of very small starches the use of a hand lens will bring out all necessary detail—a device which would, of course, result in an unpleasant effect had the ordinary process-block been adopted. The magnification is, in all cases, 150 times linear, thus facilitating comparison. The author has discarded glycerol and camphor-water for making his permanent slides, and finds that Farrant's gum, used with certain precautions, answers best.

he new edition also contains illustrations of some prepared food-stuffs, of which
is the principal constituent; but a somewhat invidious choice of certain
ietary articles has been made, one of which, although stated in the text to be
, does not figure among the illustrations.

s impossible to speak in complimentary terms of the letterpress, which con-
erous orthographical errors, and in some cases, notably under the headings
" Wheat" and "Ground-Nut," has been made the vehicle for some curious
irrelevant expressions of opinion. This is the more to be wondered at since it is
ted in the preface that the author has received valuable assistance from Pr
ch, of the Royal Agricultural College, Cirencester, in revising his proof-shee
vever, it is by the illustrations that a book of this class is to be judged, and,
ady stated above, these leave little to be desired.

H. F. E. HULTO

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE DETERMINATION OF CODEINE IN OPIUM.

By ALBERT E. ANDREWS.

THE only published methods for the estimation of codeine in opium appear to be those of P. van der Wielen (*Pharm. Zeit.*, 1903, **48**, 267) and of Caspari (*Apoth. Zeit.*, 1904, **19**, 874). These are both unsatisfactory, for in van der Wielen's method the separation of codeine and narcotine depends on the complete crystallisation of the latter from an alcoholic solution of the mixed crude alkaloids, and in Caspari's method there is loss of codeine owing to its being carried down with morphine when this alkaloid is precipitated by ammonia from an aqueous liquid containing the two alkaloids. It may not be out of place to mention here that this fact does not seem to be recognised, for to it must be attributed the presence of codeine in the morphine salts met with in commerce.

The following method is the outcome of much experimental work, and has been employed in a very large number of analyses of opium carried out in the Scientific and Technical Department of the Imperial Institute.

The principal steps involved in the process are—

(1) Preparation of an aqueous extract of the opium and the addition of lead acetate solution to precipitate colouring matters, etc., and also the narcotine. (2) The addition of sodium salicylate solution to further separate resinous bodies and also thebaine. (3) The concentration of the resulting acid aqueous liquid and removal from it of ether-soluble substances. (4) The addition of a large excess of caustic soda solution to the purified aqueous liquid so as to fix the morphine and enable the codeine to be extracted with ether.

The successful carrying out of these operations depends on certain working conditions being complied with, and for this reason it is important to give the following detailed account of the method: Twelve grms. of dry opium are completely extracted with small quantities of cold water, the total filtered liquid being made to occupy 100 c.c.; 20 c.c. of a 20 per cent. solution of lead acetate are now added, and after standing over night the liquid is filtered, preferably with the aid of a water exhaust pump, through a filter paper placed in a Buchner funnel. One hundred c.c. of the filtrate representing 10 grms. of the opium are subjected to a current of sulphuretted hydrogen and the precipitated lead sulphide is filtered off, the filtrate being collected in a 200 c.c. Erlenmeyer flask and subjected to a current of air to remove the sulphuretted hydrogen. The precipitate is washed with water, the washings being collected apart and concentrated before adding them to the main liquid, since the total filtrate should not exceed about 130 c.c. in volume. The reason for evaporating the washings separately is to avoid heating the main liquid, which would thereby become considerably coloured. Twenty c.c. of a 20 per cent. solution of sodium salicylate are now added to this liquid, the flask is corked and the contents well agitated. A resinous precipitate quickly aggregates, and the liquid is then passed through a small fluted filter-paper into a beaker, into which a few crystals of thebaine salicylate are introduced. The liquid is then well stirred, and the sides of the beaker scratched with a glass rod from time to time to facilitate the separation of any thebaine as salicylate. After standing overnight the liquid is passed through the same filter, and the above treatment is repeated until no further deposition of solid matter takes place. The liquid is now finally filtered through the same filter-paper, which is then washed with a little water. The total filtrate is now concentrated over a water-bath to about 10 to 15 c.c., and whilst still warm transferred to a glass separator, using a little water to rinse out the dish, the final rinsings being transferred to a second separator. The main liquid in the separator is now freed from ether-soluble substances by agitating well with ether, which is then separated and transferred to the second separator and shaken with the aqueous rinsings contained in it. This washing of the aqueous liquid with ether is repeated at least three times, the ether each time being passed through the second separator. The aqueous portion in the second separator is now added to the main liquid in the other separator, and 10 c.c. of a 20 per cent. solution of caustic soda are added; the strongly alkaline liquid is now extracted with ether at least four times, using for each extraction a volume of solvent rather more than the aqueous alkaline liquid. Each portion of ether is in turn passed into a second separator and washed with 20 to 30 c.c. of water, then transferred to a dry flask and dried by the use of a little anhydrous sodium sulphate before being distilled. The total ether used for the extraction of the codeine is distilled in a flask down to a few c.c., which are then allowed to evaporate spontaneously, when, as a rule, the codeine separates in well-defined crystals in a nearly pure condition.

The alkaloid is then dried, preferably in an exhausted desiccator and weighed, the weight being checked by dissolving in excess of $\frac{N}{10}$ acid and titrating the excess with $\frac{N}{10}$ caustic soda solution, using litmus or methyl red as indicator. The volumetric result is usually a little lower than the gravimetric figure, and is considered the more reliable.

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COMPOSITION OF PARA RUBBER-SEED OIL.

By SAMUEL S. PICKLES, D.Sc. and WILLIAM P. HAYWORTH, F.I.C.

The increase in the cultivation of the Para rubber-tree (*Hevea brasiliensis*) during recent years and the consequent production in large quantities of the seed has suggested many inquiries regarding the possibility of utilising the oil contained in the seed. The subject of the industrial application of this by-product has been dealt with in the current issue of the *Bulletin of the Imperial Institute* (1911, **9**, 35), and attention is there drawn to the fact that the seeds yield a liquid drying oil very similar in properties to linseed oil and capable, like the latter, of being used in the manufacture of paints, varnishes, rubber substitutes, oil-cloth, soft soap, etc. The kernels of Para rubber-seed were first examined at the Imperial Institute in 1902, and were found to contain a fixed oil which had marked drying properties (*Bull. Imp. Inst.*, 1903, **1**, 156). Since then various samples of the seed and oil have been received from time to time. In some cases the seeds had been decorticated previous to exportation, and perhaps also crushed, bruised, and otherwise damaged. Such treatment results in the partial hydrolysis of the oil and the production of an abnormal product, the reason being that the seed kernels contain a lipase, which has the property of hydrolysing the oil, and which is liberated when the cells containing it are crushed (Dunstan, *Proc. Chem. Soc.*, 1907, **23**, 168). The oil, of which the composition is here given, was extracted in this country from the kernels of undecorticated seeds. The kernels contained 48·8 per cent. of oil, which was pale yellow in colour, liquid at ordinary temperatures, and dried to a hard varnish in about twelve days on exposure to air.

On examination the oil was found to have the following constants:

Sp. gr. at 15°/15° C.	0·9239
Acid value	29·9
Saponification value	185·6
Iodine value	133·3
Titer test (fatty acids)	33°
Hehner value	96·4
Reichert-Meissl value	0·5

Examination of the Fatty Acids.—The liquid and solid (unsaturated and saturated) acids were separated by the usual method by taking advantage of the different solubilities of their lead salts in ether. The mixed fatty acids consist approximately of (1) unsaturated liquid acids 86·0 per cent., and (2) saturated solid acids 14·0 per cent. The mixed unsaturated acids form a brown oily liquid, having an iodine value = 154·2. The saturated acids were obtained as a hard, white cake, melting-point = 55° C.

Saturated Acids.—The mixed solid acids were fractionally crystallised from alcohol, and, after several crystallisations, were finally separated into two main fractions, both of which were crystalline.

Fraction 1, melting-point 69° C. This acid crystallised in shining plates, and was apparently pure stearic acid. On analysis 0·1742 grm. gave 0·4682 grm. CO_2, and 0·1995 grm. H_2O.

Whence C = 76·1 per cent. and H = 12·7 per cent.; $C_{18}H_{36}O_2$ (stearic acid) requires C = 76·0 per cent. and H = 12·7 per cent.

Fraction 2, melting-point 56·5° C. This acid or mixture of acids also crystallised in shining plates. On combustion the following results were obtained: 0·1710 grm. gave 0·4720 CO_2, and 0·1915 grm. H_2O.

Whence C = 75·3 per cent. and H = 12·5 per cent.; $C_{17}H_{34}O_2$ requires C = 75·5 per cent., H = 12·6 per cent.; $C_{16}H_{32}O_2$ (palmitic acid) C = 75·0 per cent., H = 12·5 per cent.

It is probable that this fraction is not a pure acid, but consists of a mixture of stearic and palmitic acids, which is extremely difficult to separate.

Unsaturated Acids.—The iodine value of the mixed liquid acids was found to be 154·2. The original oil was examined for the presence of linolenic esters, but on estimation it was found to yield only 6·66 per cent. of solid hexa-bromides, corresponding to not more than 2·5 per cent. of linolenic acid. It therefore seemed probable that the liquid acids consisted mainly of a mixture of oleic and linoleic acids, and confirmation was obtained on examining the products obtained from them on oxidation.

About 6 grms. of the mixture of unsaturated acids were oxidised with alkaline potassium permanganate, and the oxidation products were separated by means of ether. The ethereal solution yielded, on evaporation, dihydroxy stearic acid, which, when recrystallised from alcohol, was obtained in hexagonal plates, melting-point 130° C. The portion insoluble in ether was extracted many times with a large quantity of boiling water, and several lots of crystals were obtained, all melting, however, at 159° to 160° C. This was found to be a tetrahydroxy stearic acid (sativic acid) formed by the oxidation of the linoleic acid. It yielded the following results on analysis: 0·1312 grm. gave 0·2987 grm. CO_2, and 0·1250 grm. H_2O; C = 62·0 per cent., H = 10·5 per cent.; sativic acid ($C_{16}H_{36}O_6$) requires C = 62·1 per cent., H = 10·3 per cent.

The melting-point 159° to 160° C. is lower than that given for sativic acid by Hazura—viz., 173° C., and accordingly oxidations were conducted on pure linoleic acid obtained from Kahlbaum. The sativic acid thus obtained, however, always melted considerably below 170° C.

SUMMARY.

From the results of this examination the composition of the mixed fatty acids from Para rubber-seed oil appears to be as follows:

(*a*) *Saturated* (*solid*) *acids* = 14 per cent., consisting of *stearic acid* (melting-point 69° C.) and an acid or mixture of acids, melting-point 56·5° C.

(*b*) *Unsaturated* (*liquid*) *acids* = 86 per cent., consisting of oleic acid, 32·6 per cent.; linoleic acid, 50·9 per cent.; linolenic acid, 2·5 per cent.

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COMPOSITION OF THE FAT FROM THE SEEDS OF LOPHIRA ALATA (BANKS).

By SAMUEL S. PICKLES, D.Sc., and WILLIAM P. HAYWORTH, F.I.C.

Lophira alata (Banks) is a tree found native in Central and West Africa. It is widely distributed in Sierra Leone and through the coastal districts of West Africa.

The fat, which is obtained from the seeds, and is known as "Niam fat," or "Meni oil" in West Africa, and "zawa" oil in the Sudan, is used by the natives for culinary purposes and also as a hair oil.

During the last few years several consignments of the seeds of *Lophira alata* have been received at the Imperial Institute for investigation from Sierra Leone and also from the Sudan. Descriptions of the seeds and particulars regarding the general characters of the oil and its value have already been given in the *Bulletin of the Imperial Institute* (1908, **6**, 243; 366). In addition, Lewkowitsch (*J. Soc. Chem. Ind.*, 1907, **26**, 1265) has examined the Sudan and Sierra Leone oils, whilst amongst other workers who have examined the oil are E. Heckel (*Ann. de l'Instit. Colon. Marseilles*, 1903, series ii., vol. i., part ii., p. 1) and E. S. Edie (*Quart. J. Inst. Comm. Research in Tropics*, 1907, **11**, 124).

The following investigation of the nature of fatty acids contained in "Niam" fat was made on the fat extracted from *Lophira alata* seeds from Sierra Leone.

The kernels were ground up, and the fat was extracted by cold percolation with light petroleum. When the extraction was complete the petroleum was recovered by distillation, and the last traces were removed first by passing a current of CO_2 through the oil, and finally by heating it to 100° C. in vacuum until constant in weight. The fat obtained on cooling was pale yellow in colour, and of the consistency of butter. It had a somewhat rancid, though not unpleasant, taste. The proportion of fat in the kernels varied in the different specimens between 40 and 43 per cent.

Constants of Fat.—Sp. gr. at 40°/40° C.	0·9016	to	0·9044
Acid value	25·9	„	48·0
Saponification value ...	180·7		194·6
Iodine value	69·8		72·5
Reichert-Meissl value ...	0·8		0·9
Titer test	47°		49°

Examination of the Fatty Acids.—The acids were obtained by saponification of the oil and were separated into saturated and unsaturated acids by taking advantage of the difference in solubility of their lead salts in ether. On recovering the acids from the lead salts, the saturated (solid) acids and unsaturated (liquid) acids were found to be present in about equal proportions.

Saturated or Solid Acids.—The solid acids, which were obtained in the form of a white cake, were fractionally crystallised from alcohol and separated into two main fractions, the first crystallising from alcohol in shining plates, melting-point 77·5° C., and the second, an amorphous fraction, melting-point 56° to 59° C.

Identification of Arachidic Acid.—The melting-point of the crystalline acid (77·5° C.) is almost identical with that given for arachidic acid—viz., 78° C. A combustion of the acid yielded the following results: 0·1730 grm. gave 0·4868 grm. CO_2 and 0·1970 grm. H_2O. Whence C = 76·8 per cent. and H = 12·7 per cent.; arachidic acid, $C_{20}H_{40}O_2$, requires C = 76·9 per cent., H = 12·8 per cent.

The acid yielded a crystalline methyl ester, melting-point 53·5° C., and an amide, melting-point 99° C. There can thus be no doubt that this substance is arachidic acid.

The second fraction was amorphous, and melted indefinitely between 55° to 59° C. Repeated recrystallisation failed to bring about a separation, although it was obviously a mixture. The acids were then converted into their ammonium salts, and the dilute aqueous solution of the ammonium salts was boiled for a short time. Slight decomposition took place, some ammonia passing off; and, on cooling, a cloudiness in the solution indicated the liberation of some fatty acid. On extracting the solution with ether and evaporating the ethereal solution, an acid was obtained which had the crystalline form and melting-point (62·5° C.) of palmitic acid.

On analysis it gave the following results: 0·1755 grm. gave 0·4815 grm. CO_2 and 0·2015 grm. H_2O. Whence C = 74·9 per cent. and H = 12·7 per cent.; palmitic acid ($C_{16}H_{32}O_2$) requires C = 75·0 per cent. and H = 12·5 per cent.

Liquid or Unsaturated Acids.—On liberation from the lead salts the unsaturated acids were obtained as a yellow oily liquid having an iodine value = 139. On treatment with bromine no hexabromides were obtained; linoleic acid therefore must be regarded as absent. The iodine value appeared to indicate a mixture of oleic acid and linoleic acid in about equal proportions, and this was found to be the case.

The presence of linoleic acid was proved by the fact that on bromination linoleic tetrabromide (melting-point 114° C.), was obtained, and also by the production of a sativic acid (melting-point 160° C.) on oxidation with potassium permanganate. Confirmatory evidence of the presence of oleic acid was obtained on examination of the products of oxidation. They contained the characteristic dihydroxystearic acid, which, after crystallisation from alcohol, was obtained in hexagonal plates melting at 130° C., identical with that obtained under similar conditions from pure oleic acid. This acid was separated from the sativic acid present by repeated extraction with ether, in which the latter is insoluble.

SUMMARY.

The composition of the mixed fatty acids from the fat of *Lophira alata* seeds is approximately as follows:

Saturated (Solid) Acids: Fifty per cent., consisting of arachidic acid (melting-point 77·5° C.), palmitic acid (melting-point 62·5° C.), and probably a lower melting acid in small quantity.

Unsaturated (Liquid) Acids: Fifty per cent., consisting of about equal parts of oleic acid and linoleic acid.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Detection of Thujone in Absinthe. H. Enz. (*Schweiz. Woch. Chem. u. Pharm.*, 1911, **49**, 337-340; through *Chem. Zentralbl.*, 1911, II., 576.)—After destruction of aldehydes by boiling with aniline phosphate, the absinthe is diluted with water to a strength of 30 per cent. by volume, and 500 c.c. of the liquid are distilled with the aid of a rectifying column, 30 to 40 cm. in length, so as to obtain 9 fractions of 25 c.c. each. Where possible, it is preferable to dilute the absinthe to 60 per cent. strength, and to separate it into fractions of 50 c.c. The very delicate test of Duparc (ANALYST, 1908, **33**, 473) shows that thujone is present in the fractions 6 and 7, which contain much water, but are free from acetaldehyde and constituents of high boiling-point, such as anisol. Since, however, various oils, such as hyssop, vervain, and savin, and also carvone and citral, give the thujone reaction, a positive result in this test is not a certain proof that the liqueur was prepared from the absinthe plant. On the other hand, the non-occurrence of the reaction proves the absence of absinthol. C. A. M.

The Chemical Behaviour and Preservative Action of Sulphites in Beer. J. L. Baker and F. E. Day. (*J. Inst. Brewing*, 1911, **17**, 465-477.)—The beer was distilled with hydrochloric acid in an atmosphere of carbonic acid, the liberated sulphurous acid being collected in a little $\frac{N}{10}$ iodine and titrated with thiosulphate. Except in the case of very acid beers, only a negligible trace of other substances were carried over capable of reducing the iodine solution, the volumetric method being frequently checked by precipitating the oxidised distillate with barium chloride. It was noticed that beers to which no addition of sulphite had been made contained on the average from 20 to 25 parts per million of sulphurous acid, due to the finings and hops. This amount was deducted in all cases from the results obtained, an untreated control beer being used for the purpose in each experiment. Samples of several different kinds of ales and stouts were treated on the laboratory scale with known amounts of potassium hydrogen sulphite and calcium sulphite, and in casks with bisulphite of lime, and with a mixture of the bisulphites of magnesium and potassium (Phylax). The rate of disappearance of the sulphurous acid was then followed at intervals for seventeen weeks, as well as changes in the acidity and attenuation. It was found that potassium bisulphite rapidly lost 25 to 30 per cent. of its sulphurous acid, whilst calcium bisulphite only lost 10 to 12 per cent. A distinctly retarding effect on acid formation was shown during the first sixteen days—an effect in some cases reversed when once the beer had become strongly acid. The after fermentation was arrested to a degree roughly proportional to the sulphite present. It was observed that the greatest loss of sulphurous acid (10 to 40 per cent.) occurred during the first ten days, and usually in the first five. The acidity was found to be retarded as much by 1 ounce to the barrel of any of the sulphite preparations as by larger quantities—in fact, with heavily treated beers, acidity, when once started, proceeded more rapidly. That some combination occurs between certain constituents of the beer and the added sulphurous acid is evidenced by the fact that direct titration of the beer with iodine gave lower values than when distillation was employed. If soda were first added in excess, and then acid more than sufficient to neutralise the soda, amounts even higher than by distillation were sometimes obtained. Experiments showed that combination of the sulphite occurs, not only with beer, but separately with maltose, alcohol (methylated spirit), and dextrin, the bodies so formed possessing considerable stability, since they are not decomposed by hydrochloric acid in the cold. A considerable amount of the sulphurous acid added as a preservative to beer is thus withdrawn from chemical activity, since it is improbable that such stable compounds possess antiseptic value. Since these compounds are decomposed by alkali and water, it is suggested that they may be of the nature of unstable sulphonates or sulphurous esters. The maximum beneficial effect on beer as regards acidity prevention is obtained by the employment of sulphite preparations in such proportions that about 50 parts per million of sulphurous acid are present; double this amount is actively prejudicial. The keeping qualities of the pale ales tested were not affected by sulphites, and it is suggested that their addition to such beers may be dispensed with advantageously.

H. F. E. H.

Detection of Fat in Beeswax, Paraffin, Spermaceti, etc. M. Wagenaar. (*Pharm. Weekblad*, 1911, **48**, 479-481.)—The material is saponified, acidified, and filtered. The filtrate is then rendered alkaline, 5 drops of cupric sulphate solution are added, the mixture is boiled, and again filtered. Should this filtrate be blue in colour, it indicates that glycerol is present, and that the material under examination contains a fatty glyceride.

Estimation of Formic Acid in Foods. H. Fincke. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 88-104.)—Further experiments show that the gravimetric mercuric chloride method recommended previously for the estimation of small quantities of formic acid (ANALYST, 1911, 103) is quite trustworthy; when only 2 or 3 mgrms. of the acid are present, the average error is 0·04 mgrm. In only a few exceptional cases is the accuracy of the method affected by the presence of other substances. It has been shown (*loc. cit.*) that the interfering action of salicylic acid may be prevented by the addition of sodium chloride, and sulphurous acid, if present, may be detected readily, and due precautions taken. Glyoxylic acid is the only other substance which is likely to be met with in foods; it is found in unripe fruits, is volatile, its calcium salt is soluble, and it reduces mercuric chloride. Before estimating the formic acid, the glyoxylic acid must be destroyed by reduction with zinc and sulphuric acid. The author finds that ordinary vinegar contains but traces, if any, of formic acid, whilst wine vinegars contain appreciable quantities of this acid (up to 3·1 mgrms. per 100 c.c.), due to the fact that the wines contained formic acid. Almost all the samples of vinegar "essence" (products containing from 50 to upwards of 80 per cent. of acetic acid) contained formic acid. For the purpose of comparison, the quantities of formic acid found are calculated as parts of the total acid; on this basis, two samples yielded less than 1 part of formic acid per 1,000 parts of total acid, five samples contained more than 10 parts, whilst twenty-one other samples contained from 1·78 to 8·94 parts. W. P. S.

Estimation of Small Quantities of Water in Lard. E. Polenske. (*Untersuch. Nahr. Genussm. Kaiserl. Gesundheitsamte,* 1911, **1**, 30-36.)—The following method is proposed for estimating the small quantities of moisture which are not infrequently present in refined lard; it depends on the fact that lard containing less than 0·45 per cent. of water melts to form a clear liquid when heated to a temperature of 95° C., but becomes turbid when cooled at temperatures varying with the proportion of water present. The semi-fluid lard is placed in a test-tube fitted with a cork carrying a thermometer and heated to a temperature of 52° C. by immersing the test-tube in a water-bath; the tube is then removed from the bath and shaken until the temperature falls to 40° C.; if the lard is clear at 52° C., and does not become turbid at 40° C., it contains less than 0·15 per cent. of water. Should it become turbid at 40° C., its water-content lies between 0·15 and 0·2 per cent. If turbid at 52° C., the lard is heated to 95° C.; a turbidity at this temperature indicates the presence of more than 0·45 per cent. of water, and the amount must be estimated by heating the lard while a current of carbon dioxide is passed through it. Should the lard be clear at 95° C., it is gradually cooled until a turbidity appears, the quantity of water present being then ascertained by reference to the following table:

Water-Content.	Temperature at which Lard becomes Turbid.
Per Cent.	° C.
0·45	95·0
0·40	90·8
0·35	85·0
0·30	75·2
0·25	64·5
0·20	53·0

Of course, if the lard is not clear at a temperature of 95° C., it should be ascertained whether the turbidity is actually due to the presence of water.

W. P. S.

Analysis of Liquorice-Juice. L. and J. Gadais. (*Bull. Soc. Chim.,* 1911 [iv.], **9**, 741-743.)—The sample should be carefully prepared, by grinding, if possible; or by slicing. The moisture is determined by drying 2 grms. at 100° C. to constant weight. The insoluble matter is found by dissolving 5 grms. of the sample in 200 c.c. of cold water, settling for twenty-four hours, decanting the clear solution, adding 100 c.c. of water, settling again for twenty-four hours, again decanting, collecting the residue on a tared filter, washing, and drying at 100° C. The insoluble matter may be incinerated. The total ash should be determined on 2 grms. of the sample. For the estimation of the glycyrrhizin, 5 grms. of sample are placed in a beaker having a mark at 50 c.c., and dissolved in hot water. The cooled solution is made up to 50 c.c., and 100 c.c. of 95 per cent. alcohol are added, with thorough stirring. The deposit is allowed to settle for twenty-four hours, the liquid is decanted off into a basin, the precipitate is collected on a small tared filter, and washed three times with 15 c.c. of dilute alcohol. When all the liquors have been collected in the basin, they are evaporated down to 25 c.c. This extract is transferred to a small tared beaker having a mark at 50 c.c., and made up to the mark; 5 c.c. of water containing 1·8 c.c. of hydrochloric acid (sp. gr. 1·18) are added. The mixture is placed aside for twelve hours so that the precipitate may settle, and the clear liquid is decanted off. The deposit is washed three times with 10 c.c. of water cooled to 2° C., 0·5 c.c. of ammonia (sp. gr. 0·922) is added, and the beaker and its contents are dried at 100° C., the glycyrrhizin being weighed as its ammonia compound. The gums, etc., insoluble in alcohol are weighed on the filter on which they were collected.

A more rapid procedure is as follows: Ten grms. of the sample are dissolved in a flask gauged at 301 c.c. and having a mark at 100 c.c. The 100 c.c. of aqueous solution are mixed with about 170 c.c. of 95 per cent. alcohol; the liquid is cooled again, and made up to 301 c.c. with alcohol. After standing for two hours 150 c.c. of the clear liquid are taken for treatment as described above. It is assumed that the volume of the precipitate is generally about 1 c.c. (*cf.* ANALYST, 1911, 409-410).

J. F. B.

Polarimetric Method for the Determination of Malic Acid, and its Application in Cane and Maple Products. P. A. Yoder. (*J. Ind. and Eng. Chem.*, 1911, **3**, 563-574.)—The author has worked out a polarimetric method for the estimation of malic acid based on the increase in its lævo-rotatory power in presence of uranium acetate. This increase, when the maximum effect is obtained, is 229-fold; it should be noted, however, that the dextro-rotation of tartaric acid is also increased by uranium salts to the extent of 28-fold. If a sufficient quantity of uranium salt be used (at least 1·25 atom of uranium per mol. of malic acid), the increased rotation is independent of the concentration of malic acid—at all events, in dilute solutions. The maximum effect is not produced in presence of a high degree of acidity or concentration of hydrogen ions; mineral acids inhibit the specific action of uranyl salts, but acetic acid may be present in moderate quantities; certain hydroxy-acids, such as citric or lactic, combine with the uranium salt, so that a larger quantity of the latter is required. The simplest method of procedure presumes the absence of interfering substances, the acids being in the free state and present only in small quantities. The concentration of malic acid should be reduced to between $\frac{N}{10}$ and $\frac{N}{5}$. To an aliquot portion of the solution methyl orange and phenolphthaleïn are added, and the liquid is titrated with $\frac{N}{10}$ potassium hydroxide until the methyl orange tint lies between that shown by $\frac{N}{5}$ acetic acid and the same acid neutralised to the extent of one-fourth by potassium hydroxide. The titration is next carried farther to the neutral point with phenolphthaleïn (total acidity). The original solution is polarised in the 200 mm. tube; this liquid is returned to the main bulk, 20 c.c. are measured out, powdered uranyl acetate is added in quantity equal to about 1 atom of uranium to every atom of replaceable hydrogen of the total acids (0·046 grm. of uranyl acetate containing 51·6 per cent. of uranium for every 1 c.c. of $\frac{N}{10}$ potassium hydroxide required for complete neutralisation). The salt is dissolved in the liquid, and sufficient potassium hydroxide is added to establish the degree of neutrality to methyl orange specified above; the solution is made up to 25 c.c., filtered if necessary, polarised, and the reading corrected for the extra dilution. From the difference between the two polarisations the malic acid is calculated on the basis that for a 1 per cent. solution of malic acid in the 200 mm. tube at 20° C. the increase in lævo-rotation with white light is 29·6° Ventzke (10·25° circular), and with sodium light 28·8° V. (9·99° circular). For temperatures other than 20° C. the reading in °V. should be multiplied by $1+0{\cdot}001\ (t-20)$. If the organic acids are not all in the free state, the quantity of uranyl salt to be added cannot be accurately deduced from the phenolphthaleïn titration, and its sufficiency must be ascertained by trial, adding small increments of fully soluble uranyl acetate to the solution contained in a tubulated polarimeter tube, calculating 0·04 c.c. increase in volume per 0·1 grm. of the salt added. By the use of a tubulated polarimeter tube the method of trial may also be used to ascertain the correct degree of acidity necessary to produce the maximum increase of rotation. A measured quantity of the solution, containing a sufficient quantity of uranium, is placed in the tube, and $\frac{N}{2}$ potassium hydroxide or acetic acid, whichever may be necessary, is added from a burette through the tubulure until the maximum effect is produced, the readings being corrected for the extra dilution. This procedure may be adopted for coloured solutions where methyl orange cannot be used.

For the estimation of malic acid when interfering substances are present—for instance, in the examination of maple or cane products—the author has worked out two processes based on the preliminary precipitation of the malic acid:

1. *Lead Acetate Method.*—Weigh out 50 grms. of syrup, or 33·3 grms. of sugar and 16·7 grms. of water; add sufficient alkali to neutralise the acidity ascertained by a separate titration. Add further water to make, with the alkali solution used, 15 c.c. Mix, add 10 c.c. of lead acetate solution (sp. gr. 1·25), then 150 c.c. of neutral 95 per cent. alcohol; shake, and set aside for three hours. Decant off the clear liquid through a suction filter, remove the filtrate, bring the rest of the liquid on the filter, and return it until it filters clear; use the clear filtrate to rinse the flask, and finally wash slightly with 75 per cent. alcohol. Return the precipitate and filter to the flask, heat with water, and fully decompose the precipitate with hydrogen sulphide, filtering and washing with the usual precautions. Concentrate the filtrate, make up in a flask to 50 c.c., and determine the malic acid as described above. For 20 c.c. of such a solution about 0·7 grm. of uranyl acetate should be ample.

2. *Barium Acetate Method.*—This is preferable for cane products, owing to the possible presence of phosphoric and sulphuric acids. Weigh out half the quantities of the sample as prescribed for the lead method; neutralise with 0·0157 grm. of powdered barium hydroxide for every 1 c.c. of $\frac{N}{10}$ alkali required by titration. Add 7·5 c.c. of a 20 per cent. solution of barium acetate, mix, and allow the ingredients to dissolve. Add 220 c.c. of 95 per cent. alcohol, warm to incipient boiling in an Erlenmeyer flask fitted with a condenser. The precipitate should settle in an hour; decant the clear liquid hot through a filter, dilute the remainder with hot alcohol (1 part of

water to 14 parts of 95 per cent. alcohol), transfer to filter, rinse with this alcohol a few times (complete washing unnecessary), return precipitate and filter to the flask, dissolve in about 20 c.c. of hot water, filter, and wash with hot water. Add 1 c.c. of $\frac{N}{5}$ acetic acid to the filtrate, concentrate to 10 c.c., add further 2 c.c. of acetic acid, transfer to a 25 c.c. flask, filtering if necessary, and determine the malic acid by the method of trial (above), adding more acetic acid until the maximum effect is obtained. Maple syrups contain 0·25 to 0·5 per cent. of malic acid, cane products only 0·01 to 0·04 per cent.

Estimation of Tartaric Acid.—The uranium method may be similarly applied for the estimation of tartaric acid. The specific rotatory power, using white light and quartz compensation, of free tartaric acid is 16·3° at 27·5° C., and that of the uranyl compound is 453°; hence the percentage of tartaric acid, using the Ventzke scale, may be calculated by the formula, $\frac{P' - P}{25{\cdot}16 \times \frac{1}{2}L}$. Mixtures of tartaric acid and malic acid may also be analysed by the uranium method, using combined formulæ, provided the total quantity of the two acids present in the solution be known in terms of normal alkali.
J. F. B.

Determination of the Specific Gravity of Very Small Quantities of Milk. A. Kreidl and E. Lenk. (*Biochem. Zeitsch.*, 1911, **35**, 166-168.)—When milk is added directly to either benzene or chloroform, none of the fat present is dissolved by the solvent, the casein appearing to form a protective colloidal coating around the fat globules. The sp. gr. of any milk may, therefore, be determined by preparing a mixture of benzene and chloroform having a sp. gr. equal to that of the milk under examination, as is ascertained by adding a drop of the milk to the mixture. If the sp. grs. of the mixture and the milk be not the same, the drop of milk will either float to the surface or sink to the bottom, according as to whether it is lighter or heavier than the mixture. When the two sp. grs. coincide, the drop of milk will remain suspended in the mixture for a considerable length of time, provided that the temperature remains the same, and no fat is dissolved from the milk even after several hours' contact. The method may be applied to the determination of the sp. gr. of sour milk, and is stated to yield trustworthy results.
W. P. S.

Estimation of Starch in Brewers' Grains. C. Kreuzer. (*Zeitsch. ges. Brauwesen*, 1911, **34**, 277-280, 290-293; through *Chem. Zentralbl.*, 1911, II., 307.)—As none of the usual methods, such as treatment with malt extract, give accurate results in the estimation of the quantity of starch remaining in brewers' grains, the following iodometric process is proposed: An average sample of the grains is ground as finely as possible in a Seck (cutting) mill, and 2·5 grms. of the powder are mixed thoroughly with 10 c.c. of water. Twenty c.c. of sulphuric acid of sp. gr. 1·7 are then added, with stirring, and, after the lapse of fifteen minutes, the mixture is rinsed into a 100 c.c. flask with sulphuric acid of sp. gr. 1·3, this acid being used to dilute the whole to a volume of 100 c.c. after 5 c.c. of an 8 per cent. phosphotungstic acid solution have been added. The contents of the flask are now shaken, filtered through a double filter, and 50 c.c. of the filtrate are cooled in a stoppered 200 c.c. flask to a temperature of 0° C. Five c.c. of $\frac{N}{100}$ iodine solution are then added, the mixture is shaken, again cooled in ice water, 100 c.c. of petroleum spirit cooled to 1° C. are added, the contents of the flask are shaken for one minute, cooled in ice water, and the excess of iodine is then estimated by titrating 50 c.c. of the petroleum spirit with $\frac{N}{100}$ thiosulphate solution, starch solution being added as an indicator towards the end of the titration. The amount of iodine which has combined with the starch in the grains may then be calculated, and from this the quantity of starch present is obtained, 1 c.c. of $\frac{N}{100}$ iodine solution corresponding with 0·025 grm. of starch. The author finds that brewers' grains of various origin contain from 0·99 to 4·80 per cent. of starch. W. P. S.

Method for the Separation of Starch and Glycogen. E. Baur and E. Polenske. (*Untersuch. Nahr. Genussm., Kaiserl. Gesundheitsamte*, 1911, 1, 25-29.)—In the ordinary method of estimating starch in sausages by treatment with alcoholic potassium hydroxide solution, the glycogen is precipitated together with the starch, and is weighed with the latter. The following method of separating these two substances is given, advantage being taken of the greater solubility of glycogen in ammonium sulphate solution: A quantity of about 0·5 grm. of the mixed starch and glycogen separated from the material under examination is dissolved in 30 c.c. of water, and 11 grms. of finely powdered ammonium sulphate are added; the starch is precipitated, and, after the lapse of thirty minutes, is collected on a filter, and washed with a solution containing 11 grms. of ammonium sulphate in 30 c.c. of water. The filtrate, 30 c.c. in volume, is diluted with five times its volume of water, and 500 c.c. of alcohol are added; the glycogen thus precipitated is collected on a filter, washed with 5 per cent. alcohol, then with absolute alcohol, dried at 100° C., and weighed. The starch precipitate is washed with 50 per cent. alcohol to remove the ammonium sulphate, then dried at 100° C., and weighed.

W. P. S.

Preparation of the Copper Solution used in Bang's Sugar Titration Method. I. Bang. (*Biochem. Zeitsch.*, 1911, **32**, 443; through *Chem. Zentralbl.*, 1911, II., 307.)—The thiocyanate solution is made by dissolving 100 grms. of potassium bicarbonate in 1,300 c.c. of water, and then adding the other salts. To this solution are added 150 c.c. of a solution containing 166·67 grms. of crystallised copper sulphate per litre (*cf.* ANALYST, 1907, **32**, 180). W. P. S.

Estimation of Theobromine and Caffeine. C. Monthule. (*Ann. Chim. anal. appl.*, 1911, **16**, 137-138.)—The author describes the method in which the mixed alkaloids are dissolved in ammonia, silver nitrate is added, and the mixture is neutralised exactly with acetic acid. A gelatinous precipitate is thus obtained which contains all the theobromine, whilst the caffeine remains in solution.

Identification of Veronal. A. Jorissen. (*J. Pharm. Chim.*, 1911, **3**, 478-481.)—Three grms. of potassium hydroxide are fused in a nickel crucible and

0·3 grm. of veronal is added slowly; after being heated for two minutes, the mixture is cooled and dissolved in 10 c.c. of water. Five c.c. of this solution are treated with a few drops of ferrous sulphate solution, allowed to stand for five minutes, and then acidified with hydrochloric acid; a blue precipitate forms gradually if the substance under examination be veronal. The remaining 5 c.c. of the solution are acidified with sulphuric acid, shaken out with ether, and the ethereal extract is evaporated; on dissolving the rancid-smelling residue in water and adding a few drops of ferric chloride solution, a yellow precipitate is formed.

Estimation of Organic Substances in Water by Means of Permanganate. H. Noll. (*Zeitsch. angew. Chem.*, 1911, **24**, 1508-1518.)—This paper relates to an investigation of the reactions taking place when organic substances are oxidised with permanganate in the presence of sulphuric acid. W. P. S.

Estimation of the Volatile Acids in Wine. K. Windisch and T. Roettgen. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 155-170.)—The authors have investigated the conditions governing the distillation of the volatile acids present in wine, and, as the result of numerous experiments with wines and aqueous solutions of various volatile organic acids, come to the conclusion that in the official (German) method for estimating these acids the total quantity of volatile acids is found in the prescribed 200 c.c. of distillate only when the wine contains not more than 0·2 grm. of the acids. It is not permissible to distil over more than 200 c.c., as the distillate will then contain considerable quantities of lactic acid. If the wine under examination contains more than 0·2 grm. per 100 c.c. of volatile acids, it should be diluted with an equal volume of water, and the estimation is carried out on 50 c.c. of the diluted solution. It is recommended that the steam used in the distillation be admitted to the distillation-flask through a tube 4 mm. in diameter the lower end of which is bent at a right angle. During the distillation the current of steam, etc., should be so regulated that the 50 c.c. of wine in the distillation flask are evaporated to 25 c.c., and maintained at this volume until the distillation is completed; the steam should be under the lowest possible pressure, and about thirty minutes taken for the distillation. At the same time, wine distillates always contain some lactic acid, the quantity depending on the amount present in the wine, the pressure of the steam, and the volume of the distillate; lactic anhydride is more volatile with steam than is lactic acid. The distillation of lactic acid is considerably decreased by the use of a still-head filled with glass beads; for instance, a solution containing 1·4825 grms. of lactic acid per 100 c.c., yielded only 0·043 grm. in 100 c.c. of distillate. Only traces of formic acid and aldehyde are formed when lactic acid is submitted to steam distillation. W. P. S.

Portuguese Standards for Wines, Vinegar, and Olive Oil. (Extract from the Official Methods of Analysis. (*Ann. Falsif.*, 1911, **4**, 399-403.)—The following limits are given for certain constituents, etc., of wine, vinegar, and olive oil:

Wine.—Total solids, not less than 20 grms. per litre in the case of red wine and 16 grms. per litre for white wine. Alcohol, 7·5 to 10 per cent. Ash, not less than

1·6 grms. per litre. Volatile acidity, not more than 2 grms. per litre, calculated as acetic acid; the difference between the total solids and the total acidity (as tartaric acid) should be not less than 13 for red wine or 11 for white wine. The ratio of the glycerol to alcohol (by weight) varies from 1 : 20 to 1 : 8·5. Wines must not contain added glycerol, sucrose, or dextrin, but the presence of traces of copper, introduced accidentally, is not considered as adulteration.

Vinegar.—Wine vinegar must contain at least 4 per cent. of acetic acid and be free from mineral acids, added organic acids, poisonous metals, empyreumatic substances, etc.

Olive Oil.—Sp. gr. at 15° C., 0·915 to 0·918; refractive index, 1·4660 to 1·4670; the free acid should be not more than 1·6 per cent.; iodine value, 75 to 85; saponification value, 182 to 202; solidifying-point, 2° to 4° C. The oil must be free from arachidic acid, and not yield reactions for sesame and cottonseed oils.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Chemistry of Anæsthetics. III. : Nitrous Oxide. C. Baskerville and R. Stevenson. (*J. Ind. Eng. Chem.*, 1911, **3**, 579-582.)—The authors give a long list of impurities which may be suspected in a cylinder of nitrous oxide, and describe the methods in use in their laboratory for the detection of each. From an examination of the contents of several cylinders supplied by American manufacturers, the authors feel justified in recommending that nitrous oxide which is to be used for anæsthetic purposes should contain at least 95 per cent. of that gas (many commercial samples contain 99 per cent.) and no solids, liquids, combustible organic matter, chlorine, or other oxides of nitrogen. The last two impurities may be tested for by slowly passing 10 litres (three hours) through silver nitrate and ferrous sulphate solutions.

None of the well-known methods for the determination of nitrous oxide in a gaseous mixture of which it is the principal constituent is quite satisfactory. Explosion with hydrogen may lead to results fully 2 per cent. below the truth. The following method is recommended: A combustion-tube 100 cm. long is packed for 70 cm. of its length with rolls of copper gauze, one end of the tube being drawn out to small bore and connected by rubber to a calcium chloride tube, and this to a second similar tube and a bubbler, to show the rate of passage of the gas. The other end of the combustion-tube is fitted with a rubber stopper which carries a tee, one limb of which connects with a source of pure dry hydrogen, the other with the sample of nitrous oxide confined over mercury in a vessel in which it can be measured and its pressure and temperature observed. Hydrogen is passed through the combustion-tube at the rate of 3 or 4 bubbles a second until the copper is completely reduced and the calcium chloride tubes have reached a constant temperature. The calcium chloride tubes are weighed, replaced in position, the current of hydrogen stopped, and about 800 c.c. of the gas sample passed through the combustion-tube at the rate of 2 bubbles per second. This requires about an hour. Hydrogen is then passed through at the rate of 3 or 4 bubbles a second until all the copper oxide is reduced and all the moisture driven over into the calcium chloride tubes, this process requiring about forty-five minutes. The combustion-tube is then ready for a second determination, to begin with the passage of the nitrous oxide through the tube. A trap is used to catch any mercury and prevent its entrance to the combustion-tube. Since this is a determination of nitrous oxide indirectly by the direct determination of the oxygen present, the water from other oxygen compounds and also the moisture present in the gas must be subtracted. In the samples examined the moisture ranged from 0·13 to 0·16 per cent., and no compounds of oxygen other than nitrous oxide were detected. Oxygen itself could be detected in every sample, but the amount was too small to estimate (less than 0·1 per cent.). G. C. J.

Direct Estimation of Creatine in Pathological Urine. G. Stanley Walpole. (*J. Physiol.*, 1911, **42**, 301-308; through *Chem. Zentralbl.*, 1911, II., 394.) —The method depends upon the fact that diacetyl reacts with creatine, but not with creatinine. Albumin, which also gives a positive reaction, must first be removed by treatment with trichloracetic acid, whilst the influence of the other constituents in the urine is eliminated by making the colorimetric estimation in the following manner: Two c.c. of the urine under examination are treated with 2 c.c. of a saturated solution of sodium carbonate and 3 drops of diacetyl solution, then diluted with water to 6 c.c., and heated for one minute in boiling water. At the same time four standards for comparison are prepared by adding 0·0 mgrm., 0·2 mgrm., 0·5 mgrm., and 0·8 mgrm., of creatine to a normal urine. After the whole of the solutions have stood for thirty minutes, they are filtered, and the urine under examination is treated with a solution of 2 c.c. of normal urine and 2 c.c. of sodium carbonate solution diluted to 6 c.c., whilst to each of the standard tubes an addition is made of 2 c.c. of the urine under examination and 2 c.c. of sodium carbonate solution diluted to 6 c.c. The diacetyl reagent is obtained by distilling 50 c.c. from a mixture of 1 grm. of dioxime with 100 c.c. of 20 per cent. sulphuric acid. The diacetyl in the distillate cannot be used after a month, owing to the formation of products which give with alkali a brown pigment interfering with the reaction. For the same reason it is necessary to avoid an excess of diacetyl. The reaction is more intense in water than in urine, probably owing to the restrictive influence of ammonium salts. There is also another substance with a retarding action. This is insoluble in ether and may be removed by means of animal charcoal.

C. A. M.

Dihydroxystearic Acid in Good and Poor Soils. O. Schreiner and E. Lathrop. (*J. Amer. Chem. Soc.*, 1911, **33**, 1412-1417.)—The frequent occurrence of dihydroxystearic acid in poor soils has led to the conclusion that it is either a principal or contributory factor in infertility, and the following method is recommended for its estimation: Five to 10 pounds of the soil are extracted for twenty-four hours with 5 to 10 litres of 2 per cent. caustic soda solution. The supernatant extract is syphoned off, acidified with dilute sulphuric acid, filtered and shaken out with ether. The ethereal extract is evaporated to small bulk, and the watery residue boiled vigorously and filtered hot through a wet filter. The filtrate containing the dihydroxystearic acid is allowed to cool and is again extracted with ether, the solution being treated as before. The aqueous solution, after evaporation to small volume, deposits the acid in star-like clusters of white plates or needles, which melt when pure at 99°. One-third of the sixty soils examined showed the presence of the acid, but only two of the "good" class contained it. Of the thirty poor soils, just half the number contained the compound, which was found in virgin soils as well as in those long cultivated. Its distribution is universal throughout the United States.

H. F. E. H.

Quantitative Estimation of Volatile Fatty Acids in Fæces. R. S. McCaughey. (*Zeits. physiol. Chem.*, 1911, **72**, 140-150; through *Chem. Zentralbl.*, 1911, II., 643.)—The vacuum steam distillation method of Welde is fairly accurate for the estimation of free volatile fatty acids if a correction be made in titrating for the error introduced by the large volume of liquid and the action of carbon dioxide. For the estimation of total acidity, however, Welde's method is not very suitable. The hydrolysable substances contained in the fæces must be removed before distilling with phosphoric acid. The author extracts the fæces with boiling alcohol, makes the alcoholic extract alkaline with N. sodium hydroxide, and evaporates it to dryness on the water-bath. The residue is taken up with water, phosphoric acid is added, and the mixture is distilled with steam under vacuum. The distillate is measured and titrated with $\frac{N}{10}$ sodium hydroxide, using phenolphthaleïn as an indicator. From the number of c.c. used is deducted the correction necessary for the presence of carbon dioxide and the volatility of the phosphoric acid. This amounts to about 1 c.c. of $\frac{N}{10}$ sodium hydroxide per 1,400 to 1,500 c.c. of distillate.

J. F. B.

Bacteriological Examination of Honey. A. Sartory and E. Moreau. (*Ann. Falsif.*, 1911, 4, 259-263.)—The following micro-organisms were found in various samples of honey: Bacteria: *Bacillus subtilis*, *B. megaterium*, *B. ærophyllus*, *Sarcina lutea*, *Micrococcus radiatus*, *Staphylococcus pyogenes*, and Friedländer's *Pneumobacillus*; moulds and yeasts: *Penicillium glaucum*, *Rhizopus nigrans*, *Saccharomyces-cerevisiæ*, *Mucor racemosus*, *Aspergillus gracilis*, and *Sterigmatocystes nigra*. A new bacillus was also discovered which yielded golden-yellow colonies on plate cultures; it liquefied gelatine, the liquid portion being covered with a yellow skin. The authors place this bacillus between *B. luteus Flügge* and *B. flavus Macé*.

Preparation of Menthylglycuronic Acid. I. Bang. (*Biochem. Zeitsch.*, 1911, **32**, 445; through *Chem. Zentralbl.*, 1911, II., 307.)—The urine of rabbits, after administration of menthol, is one-half saturated with ammonium sulphate, boiled, and filtered. Ammonium menthylglycuronate separates out quantitatively on cooling the filtrate, and the acid itself may be obtained by treating the ammonium salt with acidified water or alcohol.

W. P. S.

Germicidal and Insecticidal Values of Different Soaps, with or without Associated Active Agents. H. C. Hamilton. (*J. Ind. and Eng. Chem.*, 1911, **3**, 582-584.)—The insecticidal value of soap alone is considerable, while its germicidal value is small. The nature of the glyceride present has some effect, since linseed-oil soap is found to have a greater germicidal action than cotton-oil soap (1 : 300 dilution of the former equal to 1 : 200 dilution of the latter, using the Rideal-Walker method). Further tests showed that cocoanut-oil soap has the highest value as a germicide, a dilution of 1 : 40 killing the *B. typhosus* (twenty-four-hour culture) at 20° C. in five minutes, while a dilution of only 1 : 20 was required in the case of castor- and olive-oil soaps to produce the same results. Insecticidal tests, using the bed-bug, *Cimex lectularius*, as the experimental organism, showed that little difference exists in the action of the potassium soaps of the different glycerides. The same soaps mixed with a high-boiling tar acid and caustic potash yielded some unexpected results, as shown in the following table. In each case the ingredients of the mixtures were present in equal proportions, and the specified dilutions are for each ingredient. The heading "coefficient" signifies the number of times the preparation can be diluted, as compared with carbolic acid:

OLIVE-OIL SOAP.

Combination.	Effective Dilution.		Coefficients.	
	Germicidal.	Insecticidal.	Germicidal.	Insecticidal.
Soap alone	1 : 20	1 : 100	0·17	5·0
Soap + cresylic acid	1 : 500	1 : 300	4·0	15·0
Soap + alkali	1 : 1100	1 : 50	9·0	2·5
Soap + cresylic acid + alkali ...	1 : 1000	1 : 300	8·3	5·0
	COCOANUT-OIL SOAP.			
Soap alone	1 40	1 : 100	0·33	5·0
Soap + cresylic acid	1 1000	1 : 100	8·3	5·0
Soap + alkali	1 1100	1 : 200	9·0	10·0
Soap + cresylic acid + alkali ...	1 1000	1 : 300	8·3	15·0
Cresylic acid + alkali	1 400	1 : 25	3·3	1·25
Alkali (KOH) alone	1 1200	1 : 25	10·0	1·25

The above results show that the *B. typhosus* is very susceptible to the germicidal action of caustic potash.

A point of importance is the effect of hydrolysis on the germicidal value of the soap solution. A freshly made dilution of 1 : 300 of an olive-oil soap with cresylic acid killed the test-organism under the above-specified conditions in less than one minute, while after standing eighteen hours the same dilution required between three and four minutes to produce this effect. A. R. T.

Detection of Poisonous Ricinus Constituents in Fodder-Stuffs. W. Mooser. (*Landw. Vers.-Stat.*, **75**, 107-134; through *Chem. Zentralbl.*, 1911, II., 638-639.)—By the intravenous injection of ricin into rabbits, the author has prepared an antiricin serum which serves as a precipitation reagent for the detection of *Ricinus* poison in fodder-stuffs, due to the presence of castor-oil seeds. Other bodies besides ricin are capable of giving a precipitate with the antiserum; for instance, arachin, a constituent of earth nut meal. But, since these bodies are soluble in alcohol, whilst ricin is insoluble in that medium, it is possible to eliminate the interfering substances. The procedure for the detection of ricin is as follows: Ten grms. of the suspected fodder are digested for twenty-four hours at 37° C. with 100 c.c. of pure glycerol, the extract is filtered and poured into about ten times its volume of a mixture of equal parts of alcohol and ether. The precipitate is filtered off, washed first with alcohol and then with ether, and dried at 37° C.; it is then stirred up with about 20 c.c. of 10 per cent. sodium chloride solution, and further digested with 100 c.c. of salt solution in the thermostat, the volume being finally made up to 200 c.c. before filtration. One c.c. of the clear filtrate is treated with 0·1 c.c. of the active serum, the latter being allowed to remain on the surface of the liquid, and the whole is placed in the thermostat for several hours. If ricin be present a precipitate or turbidity is produced. Only the poisonous principles of the castor-oil seed show the reaction; the presence of the harmless constituents has no effect. Meissner's statement that the poisonous principle of the seeds becomes inactive under the action of moist heat at 90° C., and is destroyed by dry heat at 130° C., is not confirmed by the authors.

J. F. B.

ORGANIC ANALYSIS.

Detection and Estimation of Methyl and Ethyl Alcohols and Formic Acid. R. F. Bacon. (*Circular, U.S. Dept. of Agriculture,* 1911.)—Substances containing small quantities of alcohol, or dilute solutions of alcohol, may be treated for the concentration of the contained spirit by distilling the material after first dissolving in them a considerable quantity of calcium or sodium chloride, and re-distilling the distillate after again adding salt. A concentration of ten times and a resulting solution containing 2 to 4 per cent. of alcohol may thus be obtained. The material should be neutralised, using phenolphthaleïn as indicator, before distilling. The sp. gr. and Zeiss immersion-refractometer reading are then determined, an agreement between these tests as to the amount of alcohol being evidence of the identity of the alcohol with ethyl alcohol. This result is confirmed by preparing the ester of paranitrobenzoic acid (Baumann-Schotten method) or of benzoic acid. To the solution a slight excess of paranitrobenzoyl chloride and an equivalent amount of sodium hydroxide are added. The liquid is shaken till the odour of the chloride disappears. The ester is a crystalline substance melting at 57° C. The benzoyl ester, prepared similarly with benzoyl chloride, is obtained quantitatively, and may be recognised by its odour and its b.-pt. (212° C.). Ethyl alcohol may be detected by the iodoform reaction and the production of ethyl benzoate. Methyl benzoate has a similar odour, but does not give the iodoform reaction.

Methyl alcohol may be detected by distilling 100 c.c. of liquid with 5 to 8 grms. of chromic acid, and testing the first 10 c.c. of distillate for formaldehyde by the usual methods. If other oxidisable substances are present the material is neutralised and one-third distilled, the distillate being oxidised as described. One part of the alcohol in 100,000 parts of water can thus be detected, and smaller amounts by previous concentration. If it is required to detect the alcohol in ethyl alcohol, the liquid is first diluted to 30 per cent. strength, and then oxidised after distillation.

Formic acid may be detected by its reduction to formaldehyde with magnesium. The solution is strongly acidified with phosphoric acid and one-third distilled. Dilute sulphuric acid and magnesium filings are added to the distillate, a vigorous evolution of hydrogen taking place. According to the amount of formic acid present, a reaction for the aldehyde will be obtainable in from five minutes to one hour. Formic acid is determined by distilling the solution containing it with phosphoric acid until the distillate is no longer acid. The distillate is evaporated after neutralisation, if necessary, to convenient volume, and an excess of platinum chloride and acetic acid equal to 1 to 2 c.c. of glacial acid for less than 1 grm. formic acid are added. The mixture is then boiled under a reflux-condenser for one hour, and the reduced platinum collected and weighed, when $Pt \times 0{\cdot}472 =$ formic acid.

A. R. T.

Separation and Estimation of Ammonia and Pyridine. M. Delépine and R. Sornet. (*Bull. Soc. Chim.*, 1911, [IV.], 9, 706-710.)—The authors have established that ammonia may be separated from pyridine by a modification of the method proposed by François for its separation from volatile amines. The ammonia is precipitated in combination with mercuric oxide, the amines remaining in the filtrate and the ammonia being liberated by distilling the mercury precipitate either with potassium iodide or with sodium thiosulphate. The mixture of ammonia and pyridine, in combination with a slight excess of hydrochloric acid, is first obtained from the sample by distillation. To this solution are added in the order named: 50 to 100 c.c. of a solution of mercuric chloride (30 grms. per litre), 30 c.c. of sodium carbonate (200 grms. per litre), 5 to 10 c.c. of sodium hydroxide (400 grms. per litre). A precipitate is formed, the liquid is made up to a known volume (500 c.c. or 1 litre) and allowed to settle. An aliquot portion (eight or nine-tenths) of the liquid is filtered rapidly, and the pyridine is distilled off in 150 c.c. of distillate, being collected in presence of hydrochloric acid. A slight excess of gold chloride is added, and the excess of the reagent, after evaporation, is removed from the pyridine aurichloride by means of anhydrous ether free from aldehyde. If evaporation be insufficient the pyridine compound yields some of its gold chloride to the ether, whereas if heating be continued too long some of the excess of gold chloride becomes insoluble in ether. Good results are obtained by evaporating the liquid to a small bulk on the water-bath and completing the evaporation by exposure in the vacuum desiccator for some days. Platinic chloride is more convenient than gold chloride, since the liquid may then be evaporated without danger. The excess of the reagent is removed by treatment in the cold with a mixture of 1 part of absolute alcohol and 3 parts of dry ether. The residue of pyridine platinichloride is incinerated.

For the estimation of the ammonia, the mercury precipitate is washed three or four times on the filter with a liquid containing 10 grms. of sodium carbonate and 10 grms. of sodium hydroxide per litre. The filter and precipitate are then transferred with water to a distillation flask, 50 c.c. of a solution of thiosulphate (200 grms. per litre) are added, 200 c.c. of distillate are collected in an excess of hydrochloric acid, and the solution is evaporated on the water-bath in a glass vessel, the residue being dried at 105° C. and weighed as ammonium chloride. J. F. B.

Estimation of Caoutchouc as Tetrabromide. D. Spence and J. C. Galletly. (*Le Caout. et la Guttapercha*, 1911, 8, 5313-5316.)—Having shown (ANALYST, 1911, 361) that the decomposition of caoutchouc tetrabromide by nitric acid involves a serious error, the authors advocate the following procedure: From 0·1 to 0·5 grm. of the tetrabromide is weighed out in a platinum crucible, and intimately mixed with 1 grm. of sodium carbonate, the amount of halogen in which has been previously determined. The whole is covered with a layer of fusion mixture sufficient to prevent any loss of hydrobromic acid during the combustion. The crucible is heated gently and gradually for half an hour up to a red heat; the contents should never be allowed to fuse completely. After cooling, the mass is dissolved in water, the solution is filtered, and the halogen is estimated gravimetrically. The combustion may be carried out equally well by using sodium carbonate alone instead of the fusion mixture. Under certain conditions the results tend to be slightly too high, owing to the presence of some silver salt which remains undecomposed if the solution to be precipitated is merely acidified with nitric acid; it is necessary, therefore, to add a considerable excess of nitric acid and to precipitate the bromide in a strongly acid solution. The caoutchouc tetrabromide should be reduced to powder in order to obtain a uniform and intimate mixture with the alkali; it is desirable that not less than 0·3 to 0·4 grm. be weighed out for analysis. A temperature corresponding to that of incipient fusion with sodium carbonate alone gives the best results in practice, this being sufficient to decompose all traces of organic matter tending to make the results too high; an excessive temperature leads to low results. J. F. B.

Estimation of Halogens in Organic Compounds. T. St. Warunis. (*Chem. Zeit.*, 1911, **35**, 906-907.)—The author describes the following method for the estimation of halogens in organic compounds: 0·2 to 0·3 grm. of the finely powdered substance is stirred up in a nickel crucible with a mixture of 10 grms. of powdered potassium hydroxide and 5 grms. of sodium peroxide, some of the mixture being placed as a covering layer on the top of the portion containing the organic substance. The crucible is covered and placed in a drying oven at a temperature not exceeding 85° C. until the contents have caked together. The crucible is then heated at first with a very small flame, the heat being increased as fusion proceeds. After the melt has cooled it is dissolved in water, and dilute nitric acid is added drop by drop until the solution is acid, care being taken to avoid a rise of temperature. The liquid is filtered, and the halogen is estimated either gravimetrically or volumetrically by means of silver nitrate. In the case of iodo compounds a little iodate may be formed, in which case it is advisable to add sulphurous acid to the solution of the melt. The method is rapid and gives accurate results in the case of solid and not very volatile halogen compounds; in the case of nitrogenous substances there appears to be no danger of the formation of cyanide. J. F. B.

Estimation of Lecithin. C. Virchow. (*Chem. Zeit.*, 1911, **35**, 913-914.)—The following method is stated to yield accurate results and to be more convenient and satisfactory in its manipulation than the methods hitherto employed: The main feature consists in the treament by which the filtration of the ethereal solution of the lecithin is accelerated; the portion of the extract, which is insoluble in ether, is no longer of a slimy consistence, and a clear filtrate is obtained. One grm. of the lecithin preparation is placed in a narrow-necked flask of about 20 c.c. capacity and boiled with three successive quantities of 10 c.c. of absolute alcohol. The alcoholic extracts are filtered into a weighed flask, and the insoluble portion is washed with alcohol so that the volume of the total filtrate is about 60 c.c.; this alcoholic extract is evaporated, the residue is dried, weighed, and then extracted with three successive quantities of 10 c.c. of ether. The ethereal solution is filtered through asbestos, the insoluble portion is washed three times with ether, and the united filtrates and washings are evaporated. After being weighed, the dry residue is dissolved in fuming nitric acid, the solution is transferred to a platinum crucible, and evaporated to dryness. One grm. of anhydrous sodium carbonate is now added, then 5 grms. of a mixture consisting of 2 parts of potassium nitrate and 1 part of crystallised sodium carbonate, and the contents of the crucible are fused. The fused mass is dissolved in water, acidified with hydrochloric acid, then neutralised with ammonia, and the phosphoric acid is estimated by the magnesia method in the usual way.

W. P. S.

Lecithin. R. Cohn. (*Zeitsch. öffentl. Chem.*, 1911, **17**, 203-217; through *Chem. Zentralbl.*, 1911, II., 304-305.)—The author has investigated the conditions governing the extraction of lecithin from substances containing it. That lecithin cannot be extracted completely by ether from yolk of egg, etc., is due to the adsorption of lecithin by the albumin. Cold alcohol extracts the whole of the lecithin, and there is no necessity to use hot alcohol, as further treatment with the latter only removes very small quantities of substances (phosphatides) which are insoluble in cold alcohol. There does not appear to be any chemical combination between the lecithin and the albumin. It is not possible to extract the whole of the lecithin by means of ether, alcohol, or other solvent, when the original material has been heated. For the estimation of lecithin in preparations containing added phosphoric or glycerophosphoric acids, the sample is extracted first with ether and then with alcohol; the alcohol extract must next be extracted with chloroform, which dissolves the lecithin from quantities of the above-mentioned acids which are found in the alcoholic extract. The nitrogen-content of the alcoholic extract should also be estimated in order to prove the actual presence of lecithin; if this substance be present alone the ratio of nitrogen to phosphorus will be approximately 1 : 1. W. P. S.

Estimation of Naphthalene in Crude Coal Gas. A. Albrecht and F. Müller. (*J. für Gasbeleuchtung*, 1911, **54**, 592-594; through *Chem. Zentralbl.*, 1911, II., 493.)—Rutten's picric acid method, which usually gives excellent results, is not suitable for crude coal gas, with which it yields too low values. With regard to other methods, experiments showed that only the purification of the gas with sulphuric acid and with alkali solution is applicable. Traces of tar left behind do not interfere with the sharpness of the change of colour. All rubber tubing employed should be old and as short as possible. Estimations of the naphthalene in the coal gas from horizontal and upright retorts indicated that the higher proportion in the latter is due to the higher temperature at which the gas leaves the retort. The three kinds of coal tried yielded gas containing practically the same amounts of naphthalene (*cf.* ANALYST, 1910, **35**, 74). C. A. M.

Characteristics of Conifer Fatty Oils. C. Grimme. (*Chem. Zeit.*, 1911, **35**, 925-927.)—With the exception of *Pinus Gerardiana*, which is a commercial product from the East Indies, the whole of the seeds examined were obtained from European plants. The results are summarised in the following table:

OILS.

Seeds of—	Yield of Oil.	Sp. Gr. at 15° C.	Solidification Point.	$[N]_D$ 35° C.	Acid Value.	Saponification Value.	Iodine Value (Wijs).	Fatty Acids.	Glycerol.	Unsaponifiable Matter.
	%		° C.					%	%	%
Pinus sylvestris	32·1	0·9326	– 28 to – 29	1·4704	0·8	189·8	147·1	91·48	10·32	2·06
P. montana	29·6	0·9318	– 25 to – 26	1·4698	0·8	189·6	145·7	91·34	10·32	2·18
P. cembra	35·7	0·9316	– 20 to – 21	1·4710	0·3	188·0	156·3	92·26	10·25	0·92
P. picea	32·8	0·9268	– 25 to – 26	1·4879	5·2	190·5	120·9	89·75	10·36	3·43
P. abies	31·6	0·9312	– 26	1·4742	0·9	192·0	120·5	91·51	10·44	1·53
P. pinea	21·8	0·9326	– 22	1·4685	4·2	192·6	120·9	91·52	10·28	1·62
P. Gerardiana	30·7	0·9307	– 17	1·4679	1·6	191·3	118·3	91·46	10·36	1·64
Cupressus sempervirens ...	10·8	0·9320	– 4	1·4857	2·3	188·6	135·1	91·58	10·30	2·06
Thuja occidentalis ...	15·0	0·9298	– 8	1·4795	2·1	186·7	154·8	89·90	10·32	3·22

FATTY ACIDS.

Seeds of—	Melting-Point.	Solidification Point.	$[N]_{D40°}$ C.	Neutralisation Value.	Iodine Value.	Mean Molecular Weight.
	° C.	° C.				
Pinus sylvestris	– 10	– 7 to – 8	1·4626	191·3	153·6	293·3
P. montana	0	– 4	1·4634	191·2	150·5	293·4
P. cembra	– 9	– 11	1·4607	189·0	158·0	296·8
P. picea	– 11 to – 13	– 15 to – 16	1·4875	192·1	121·6	298·8
P. abies	– 12 to – 16	– 17 to – 19	1·4672	190·4	121·8	301·5
P. pinea	– 15 to – 16	– 19	1·4636	194·3	120·8	288·7
P. Gerardiana	0	– 3	1·4613	196·7	125·0	285·2
Cupressus sempervirens ...	4 to 5	1	1·4795	193·0	142·1	290·7
Thuja occidentalis ...	– 3	– 7 to – 8	1·4736	185·7	155·7	302·1

All the oils had aromatic odours and possessed strong drying properties. The oil from *P. cembra* contained palmitic, oleic, linolic, and hydroxy acids, with traces of myristic acid. The oils of *P. picea* and *P. abies* are known in commerce as pitch-tree oil and pinaster (or red pine-seed) oil respectively. C. A. M.

Examination of Coprah. (*Chem. Rev. Fett- u. Harz-Ind.*, 1911, **18**, 195-196.)—In order to obtain uniformity in the analytical results the following process of analysis has been agreed upon and tested by the Association of German Oil-Millers: The commercial samples of coprah, weighing about 2 kilos., are broken in half, and only half of each used for the analysis. These portions are broken by hand into pieces about the size of a nut, which are then crushed in a mill so constructed that it yields small fragments without causing any separation of oil. About 500 grms. of the thoroughly mixed material are now introduced into a flask provided with a ground-in stopper. Ten grms. are weighed and placed in a filter-paper cartridge, any oil that exudes during the weighing being taken up with a plug of cotton-wool, which is also introduced into the cartridge. The latter is extracted beneath a reflux condenser with petroleum spirit (with a boiling-point up to 60° C.) in a Soxhlet apparatus, the highest point of the siphon tube of which is only 1 cm. above the top of the cartridge. The extraction is continued for four hours, the temperature of the water-bath being meanwhile not allowed to exceed 75° C., after which the cartridge is removed from the apparatus, and, after expression of as much of the solvent as possible by means of a glass rod, is dried for half an hour in a hot-water oven. Its contents are then ground up with quartz sand, replaced in the cartridge, and again extracted for two hours. Provided that the material has been sufficiently finely ground, a second grinding-up and third extraction are unnecessary. The solvent is evaporated from the united extracts, and the residue of oil dried in the oven at 105° to 106° C. and weighed. C. A. M.

[The essential part of this process appears to be the fine grinding of the sample after the bulk of the oil has been extracted by the solvent and completion of the process by further extraction of the fine powder. The necessity for this has long been recognised in England, and is enjoined for the analysis of all agricultural feeding stuffs in the official regulations of the English Board of Agriculture and Fisheries.—Ed.]

Neats-Foot Oil. **W. Fahrion.** (*Collegium*, 1911, 209-212; through *Chem. Zentralbl.*, 1911, II., 397-398.)—Eleven samples of commercial neats-foot oil and five samples of bone oil gave the following values:

	Acid Value.	Saponification Value.	Hehner Value.	Iodine Value.	Melting-Point of Fatty Acids.	Mean Molecular Weight of Fatty Acids.
					° C.	
Neats-foot oil ...	2·4 to 10	189 to 197·6	94·7 to 95·6	59·7 to 85·2	21 to 37	275·4 to 288·6
Bone oil ...	2·4 to 9	186·7 to 196·1	95 and 95·7	67·4 to 79·8	13 to 34	279·0 to 294·8

Since the fluidity in the presence of cold is reduced by a rise in the acid value, the author advocates the adoption of 6 (= 3 per cent. of oleic acid) as the highest limit for the acid value of neats-foot oil. The saponification value recorded by different observers for pure oils ranges from 194 to 199, and only in the case of commercial samples does it fall as low as 189. Holde and Stange found ten samples with iodine values of 78·3 to 85·2 to be adulterated. This value may be raised a few units by subjecting the oil to cold. The mean molecular weight of the fatty acids ought not to be outside the limits of 275 and 284, and their melting-point should be as low as possible. For the detection of vegetable oils, valuable information is afforded by a determination of the iodine value of the liquid fatty acids. Thus, three samples of neats-foot oil gave liquid fatty acids with iodine values of 94·9, 96·9, and 92·7, whilst in the case of one which was undoubtedly adulterated the corresponding value was 105·4.

C. A. M.

Estimation of Nitric Nitrogen in Gun Cotton, Nitroglycerine, and the Like. H. Pellet. (*Ann. Chim. anal. appl.*, 1911, **16**, 294-296.)—In handling nitroglycerine and some other products it is necessary to proceed rather differently to Schloesing's original method in order to avoid too rapid action. The acid solution of ferrous sulphate is boiled in the flask to expel air from the apparatus, and the nitroglycerine in more or less dilute solution is then added at any desired rate. (*Cf.* ANALYST, 1911, 243.)

G. C. J.

Composition of Soya-Bean Oil. S. Keimatsu. (*Chem. Zeit.*, 1911, **35**, 839-840.)—The oil examined gave the following analytical values: Sp. gr. at 15° C., 0·9265; viscosity (Engler) at 20° C., 8·9 to 9·0; melting-point, −7° to −8° C.; solidification-point, −15° to −16° C.; saponification value, 190; iodine value, 132 to 135; melting-point of fatty acids, 23° to 24° C.; and solidification-point of fatty acids, 16° to 17° C. It contained about 12 per cent. of saturated fatty acids, chiefly stearic and palmitic acids, and about 80 per cent. of unsaturated fatty acids, about half of which consisted of an acid isomeric with linolic acid, and yielding a hydroxy acid melting at 158° to 159° C. They also contained ordinary linolic acid (yielding satavic acid melting at 173° to 175° C.) and oleic acid (with a dihydroxy acid melting at 126° to 127° C.). Together these two acids formed about 15 per cent. of the unsaturated fatty acids. The unsaponifiable matter contained 0·2 per cent. of phytosterol, in which no stigmasterol could be detected. (*Cf.* ANALYST, 1911, 358.)

C. A. M.

Detection of Hydrocarbon and Resin Oils in other Oils. A. E. Outerbridge, jun. (*Chem. Eng.*, 1911, **14**, 347-348.)—The author has observed that both fluorescent and de-bloomed mineral oils, and also resin oils, are so affected by the ordinary enclosed electric-arc light as to have this fluorescence enormously increased, rendering it possible to detect the presence of these adulterants in linseed or other non-fluorescent oils, even if present to the extent of only 0·1 per cent. If the plain glass cover of this light fits well, the air inside is rarefied and a faint reddish light is given out. In the path of these rays the above phenomena are produced. Standards may be prepared and the amount of adulteration of non-fluorescent oils determined by means of the fluorescent scale thus obtained.

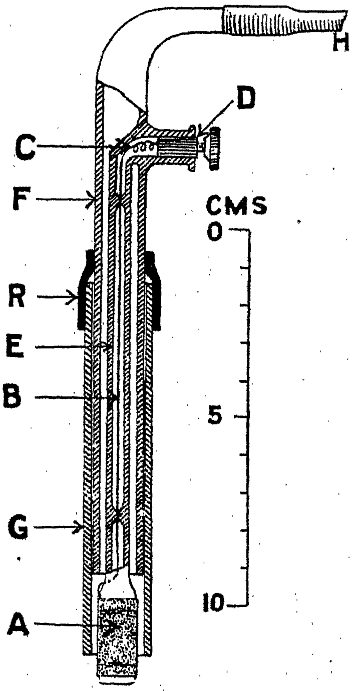
A. R. T.

Estimation of Ketones in Essential Oils. E. K. Nelson. (*J. Ind. and Eng. Chem.* 1911, **3**, 588-589.)—One to 2 grms. of the substance are heated in a water-bath under a reflux condenser with 35 c.c. of hydroxylamine reagent (prepared by dissolving 20 grms. hydroxylamine hydrochloride in 30 c.c. water, and adding 125 c.c. of alcohol free from aldehydes) and 2 grms. of sodium bicarbonate, and the liquid cooled, made acid by the addition of 6 c.c. of strong hydrochloric acid through the condenser, and the whole diluted with water to 500 c.c. The solution is filtered, and an aliquot part of the solution neutralised with $\frac{N}{2}$ caustic soda to methyl-orange. The excess of hydroxylamine is then titrated with $\frac{N}{10}$ caustic soda and phenolphthaleïn. The results are fairly satisfactory, showing a percentage equal to about 96 to 102, as compared with theory, with most ketones except fenchone, with which low results (86 per cent.) were obtained. The method serves for the assay of oils containing carvone, camphor, pulegone, and thujone as the chief constituent. (*Cf.* H. E. Burgess, ANALYST, 1903, **29**, 78.) A. R. T.

Use of Triketohydrindene Hydrate for the Detection of Proteins and their Derivatives. E. Abderhalden and H. Schmidt. (*Zeitsch. physiol. Chem.*, 1911, **72**, 37-43; through *Chem. Zentralbl.*, 1911, II., 640-641.)—Triketohydrindene hydrate (Ruhemann, *J. Chem. Soc.*, 1910, **97**, 2025) gives a blue coloration with α-amino acids, polypeptides, peptones and proteins. Exceptions are—proline, hydroxyproline, and pyrrolidone carboxylic acid. A blue coloration is only formed when a free amino group and a free carboxyl group are present simultaneously. For carrying out the test a solution of 0·1 grm. of the reagent in 300 c.c. of water is employed; 1 to 2 drops of this reagent are added to 1 c.c. of the liquid to be tested, and the mixture is heated to boiling. The liquid to be tested must have a neutral reaction. Glycocoll and alanine show a perceptible reaction at a dilution of 1 : 10,000; tyrosine at 1 : 5,000. Urine always gives a positive reaction; blood, from which the proteins have been removed by ferric hydroxide, reacts in nearly all cases, owing to the presence of products of proteolysis. By means of the method of titration in presence of formaldehyde, the authors have been able to prove the presence of amino nitrogen in blood deprived of its proteins; amino acids could not, however, be isolated. J. F. B.

An Inner Anhydride Reaction of Proteins. B. Bardach. (*Chem. Zeit.* 1911, **35**, 934.)—A reaction for inner anhydrides and groups capable of forming anhydrides has been described previously by the author (ANALYST, 1909, **34**, 326; 1911, 458). The fine, needle-like crystals obtained in the test consist of pure iodoform; proteins, such as peptone, gelatin, etc., possessing the property of causing iodoform to crystallise from its solution in the form of needles instead of the usual hexagonal prisms. This change in the crystalline form of iodoform by proteins takes place when an alcoholic solution of iodoform is mixed with a protein solution, and also when the protein has been treated previously with iodine. The effect on the iodoform is not due to the colloidal nature of the proteins, as colloidal solutions of silica, sulphur, and starch do not behave in a similar way, but is probably owing to the presence of the complex—CO—O—in the protein molecule. In general, the reaction may be used for the detection of aromatic inner anhydrides, aromatic hydroxy acids with long side-chains, and aromatic polyhydroxy-ketones. It is possible to distinguish these hydroxy-acids from phenols by using the test in conjunction with Millon's reagent; a positive result with the latter and a negative reaction for anhydrides indicates the presence of phenols. W. P. S.

Electrometric Method for the Estimation of the Acidity of Tan Liquors. Part II. J. T. Wood, H. J. S. Sand, and D. J. Law. (*J. Soc. Chem. Ind.*, 1911, **30**, 872-877.)—In the first description of this method (ANALYST, 1911, 121) several details were left open, notably the form of hydrogen electrode most suitable for the purpose. After trying other forms, the authors recommend the electrode here illustrated, which combines rigidity with rapidity of saturation and ease of manipulation.



The platinum plate consists of a piece of very thin foil which is wrapped round a cylindrical body of glass, A, and is held to the latter by some platinum wire, which passes through the glass and up the stem, E, where it is continued in the copper wire, B, the platinum wire, C, and finally in the terminal D. The stem, E, is fused in the manner depicted into a wider glass tube F which can be connected to the rubber supply-tube, H, for the hydrogen. The tube, F, ends a short way above the platinum foil, and over it is slipped the tube, G, which is just wide enough to allow it to slide up and down F readily. A piece of thick-walled rubber tube, R, holds G firmly, and F sufficiently to keep G in position without hindering it from being pushed up and down when desired. During the operation of platinising, G is pushed up completely. While the electrode is in use, G is adjusted to be a few mm. above the bottom of the foil, thus allowing the latter to dip into the liquid by this amount, whereas during the time the electrode is left filled with hydrogen when not in use, G is pulled down as far as possible, and at the same time the rubber delivery-tube, H, is clipped as close to the electrode as possible. The dimensions of the cylinder of platinum foil are 16 to 20 mm. high by 11 mm. diameter. The electrode is conveniently suspended from a ring fixed to the burette-stand by a piece of string, so as to be a few mm. above the bottom of the beaker containing the liquor to be titrated, which is then stirred by hand by the electrode itself. A solution of gallotannic acid may be neutralised completely with $\frac{N}{10}$ alkali without any appreciable darkening when the hydrogen electrode is employed, as the escaping gas hinders oxidation, whereas oxidation and darkening always occur at the neutral point during ordinary titration and the degree of oxidation affects the consumption of alkali. Unlike ordinary indicators, the hydrogen electrode is unaffected by large quantities of protein matter such as may be present in tan liquors.

The voltmeter attached to the potentiometer box gives readings to one-fifth centivolt. Seeing that other sources of error, such as contact potential, are greater than the error of reading the potentiometer, this is quicker and more convenient than the method in which the slide wire is used. Hydrogen voltages determined for various pure acids occurring in tan liquors are given, and the differences between calculated and experimentally determined values do not exceed 1 centivolt in any case except that of lactic acid, where the difference is 2·1 centivolts. Noyes, in his paper on the theory of indicators (ANALYST, 1910, **35**, 415), recommends that the ratio between the hydrion concentration corresponding to the correct neutralisation of an acid and that shown by the indicator employed in the titration should, if possible, be kept within the limits 3·5 and 1/3·5. Translating this into terms of the electrometric method, this would require an accuracy of ± 3·1 centivolt, which it is shown is attainable. G. C. J.

Use of Nickel Hydroxide in Tannin Estimation. P. Singh. (*J. Soc. Chem. Ind.*, 1911, **30**, 936-937.)—In a preliminary note the author proposes the substitution of nickel hydroxide for hide-powder in tannin determinations. The preliminary operations are carried out as in the hide-powder method. One hundred c.c. of the optically-clear tannin liquor are shaken with about 100 c.c. of a paste-like mass of freshly prepared nickel hydroxide (washed free from alkali and sulphate) made by suspending this reagent in water. After thorough shaking, the mixture is made up to 250 c.c., and filtered, when the filtrate is quite clear and gives no tannin reaction. Fifty c.c. of this filtrate are then evaporated, dried, and the residue weighed. The non-tannins are calculated on 100 c.c. of the original liquor. Experiments show that the reagent can be employed with all kinds of tanning materials. A large number of analyses are given, and the results shown in comparison with the standard (hide-powder) method. A. R. T.

INORGANIC ANALYSIS.

Some Uses of Amalgamated Aluminium in Chemical Analysis. E. Kohn-Abrest. (*Bull. Assoc. Chim. Sucr. Dist.*, 1911, **28**, 938-943; through *Chem. Zentralbl.*, 1911, II., 302.)—Tannin is precipitated from its solution by the action of aluminium slightly coated (amalgamated) with mercury, and dark-coloured oak bark extracts, etc., may be decolorised completely by the same means. Five grms. of aluminium, which have been immersed for three minutes in a 0·5 per cent. mercuric chloride solution and then washed are employed for every 200 c.c. of the solution to be treated. W. P. S.

Elementary Analysis. A. Kurtenacker. (*Zeitsch. anal. Chem.*, 1911, **50**, 548-565.)—Several metallic oxides were tried in substitution for the copper oxide of the ordinary combustion tube. Of these, only cobalt oxide was found as satisfactory as copper oxide, and the author prefers it to copper oxide because of the greater speed and lower temperature at which a combustion may be carried out by means of it. To prepare the oxide in suitable form, cobalt nitrate is first dehydrated and then decomposed by gradually raising it to a bright red heat. The porous mass is easily broken to a convenient size. It is somewhat hygroscopic. It is said that a combustion may be made with so short a layer as 10 cm. of this material. Nickel oxide can be used, but has marked disadvantages as compared with cobalt oxide, whilst no satisfactory results could be obtained when using ferric oxide, oxide of manganese, tungstic or molybdic acid.

Metallic nickel, placed at the forward end of a combustion-tube, serves well for the reduction of oxides of nitrogen, but the form of the metal is important. A spiral of ordinary thin sheet is useless. Nickel oxide is first made by igniting the nitrate, and the oxide is then reduced in a current of hydrogen at not too high a temperature. A porcelain boat, 14 cm. in length, is filled with this reduced nickel and maintained at a scarcely visible heat in the forward part of the combustion-tube. Under such conditions, metallic nickel has a scarcely measurable reducing action on the carbon dioxide passing over it in the course of an ordinary combustion. But in a nitrogen estimation by Dumas' method, the error may be appreciable, and therefore, still further forward in the tube, a short boat containing cobalt oxide is placed and maintained at something less than a red heat. G. C. J.

Qualitative Analysis without the Use of Hydrogen Sulphide. E. Ebler. (*Zeitsch. anal. Chem.*, 1911, **50**, 603-614.)—In the method of analysis without the use of hydrogen sulphide previously described by the author (*Zeitsch. anorg. Chem.*, 1905, **48**, 61; *Zeitsch. anal. Chem.*, 1908, **47**, 555), the removal of arsenic at an early stage, to prevent its coming down as an insoluble arsenate of magnesium or other metal, was accomplished by a rather troublesome process. It is now recommended that the separation of arsenic should be the first operation of analysis, and that it should be accomplished by distillation with hydrochloric acid and hydrazine bromide (ANALYST, 1910, **35**, 324).

The decomposition of complex cyanides by means of sulphuric acid is not recommended, as certain metallic chlorides may be volatilised and insoluble substances of the type of chrome ironstone may be formed. It is better to effect the decomposition by boiling for half an hour with equal parts of fuming nitric and concentrated hydrochloric acid under a reflux condenser. If an insoluble residue remains, it may contain cyanogen, but the solution is free from complex cyanogen compounds and from cyanide. Any insoluble residue may now be subjected to fusion with potassium bisulphate without risk of loss or formation of insoluble substances, since mercury, tin, antimony, and chromium will have passed into the aqua regia solution, unless the chromium was originally present as chrome ironstone. The aqua regia solution is evaporated with more nitric acid until the hydrochloric acid is wholly decomposed, and the residue is taken up in dilute nitric acid, and the insoluble matter, if any, filtered off and washed. The solution is then examined by the methods described in the earlier papers.

The insoluble residue may contain lead chloride or sulphate, complex cyanides (of iron, copper, nickel, cobalt, zinc, etc.), silver halides, stannic and antimonic acids, tin phosphate, silica, and the sulphates of the alkali earth metals. Lead is first extracted by means of a hot nitric acid solution of ammonium nitrate, which does not remove any silver or antimony. A bisulphate fusion follows, and the solution of the melt is either analysed separately or mixed with the nitric acid solution obtained at an earlier stage. The silver in the insoluble residue is next reduced with zinc after grinding to a cream with water and adding dilute sulphuric acid. The silver and other insoluble matter is washed free from chloride, and the silver and excess of zinc are then dissolved in hot dilute nitric acid. Reduction with zinc is preferred to extraction with ammonia or cyanide, as it avoids risk of solution of tin, antimony, and silica. The residue, freed from silver, is fused at a low temperature with alkali and sulphur, and, in the filtered solution of the melt, tin and antimony are precipitated by addition of acid. The sulphides are filtered off, and the filtrate, which may contain phosphoric acid and silica, is boiled to expel hydrogen sulphide, filtered from sulphur, and preserved. The residue from the extraction of the sulphur fusion is fused with sodium carbonate, and the aqueous extract of the melt is filtered from the carbonates of the alkali earth metals and mixed with the filtrate from the sulphides of tin and antimony. The mixed filtrates are tested for the presence of phosphoric acid, and evaporated to dryness to render silica insoluble. G. C. J.

New Method for Separation of Cerium. C. James and L. A. Pratt. (*J. Amer. Chem. Soc.*, 1911, **33**, 1326-1330.)—The precipitation of cerium as basic ceric nitrate may be made almost quantitative by the addition of potassium bromate to the faintly acid or neutral solution of the rare earth nitrates. The nearly neutralised solution is boiled with potassium bromate in presence of a lump of marble, which is found to be the best substance for keeping the solution neutral. Powdered marble must not be used, nor must boiling be too prolonged, or the reaction will become distinctly alkaline, and the precipitate will be contaminated by didymium, if present in the solution. When a small quantity of the supernatant liquid, withdrawn for the purpose, gives no reaction with hydrogen peroxide, the lump of marble is removed, and the precipitate is filtered off and washed with a 5 per cent. solution of ammonium nitrate, since attempts to wash with water lead to the formation of colloidal sols. The filter-paper and precipitate are returned to the beaker and treated with nitric acid and a little potassium bromate, to oxidise the paper. The paper must be completely oxidised. After evaporation to expel acid, the residue is dissolved in water and treated as before with potassium bromate and marble. After removal of the marble, filtering and washing, the precipitate is redissolved in the original beaker by means of hydrochloric acid, the solution filtered and evaporated to expel acid, and cerium finally precipitated as oxalate and weighed as CeO_2. The error, usually positive when the above directions are followed, should not exceed 0·8 per cent. By a modification of the process many kilograms of cerium salts may be prepared in a few hours, and of such a degree of purity that saturated solutions of the nitrate show no absorption bands when observed in a layer 20 cm. thick. G. C. J.

Accurate Technical Estimation of Calorific Value of Anthracite. A. G. Blakeley and E. M. Chance. (*J. Ind. Eng. Chem.*, 1911, **3**, 557-559.)—The authors have found it possible to dispense with the use of soft coal or other kindler in determinations of the calorific power of anthracite, even when this contains as much as 40 per cent. of ash. The difficulty in securing the complete combustion of anthracite was found to depend mainly on the chilling of the coal by the rapid conduction of heat through the nickel capsule and heavy platinum wire of the Atwater calorimeter employed by the authors. This can be countered and complete combustion insured by the simple expedient of placing in the capsule, below the coal, a pinch of soft asbestos, which is then lightly tamped in place by some blunt instrument, so as to present a concave depression, with the centre perhaps 2 mm. lower than the edges. The mat at its thinnest point, the centre, should not be less than 2 mm. thick. The coal should at least pass a screen of 80 meshes to the inch, and the usual routine of combustion is pursued, except that a pressure of oxygen of 25 atmospheres is used. G. C. J.

Determination of Sulphur in Coal by Means of Jackson's Candle Turbidimeter. H. F. Muer. (*J. Ind. Eng. Chem.*, 1911, 3, 553-557.)—Jackson's turbidimeter (*Techn. Quarterly*, 1900, **13**, 278) consists of a glass cylinder about 28 mm. in diameter and 25 cm. high, graduated throughout its height in mm., and supported 75 cm. above the top of a carriage candle-holder, which carries a standard candle. Originally designed for measuring the turbidity of water, it has been adopted by the United States Geological Survey for field use in determining sulphate and lime in waters from the turbidity produced by barium chloride and ammonium oxalate respectively. Attempts to make use of it for the determination of sulphur in coal by precipitating the rinsings of a Mahler bomb with barium chloride, at the conclusion of a calorimetric determination, led to results which did not accord well with gravimetric results or with each other. It was found that nitrates in the solution, in quantities such as are found in coal analysis, did not affect the results, nor were variations in the size of the flame or of the distance of the top of the candle-tube from the bottom of the glass-tube of importance, though the flame should not much exceed 40 mm. in height if the deposition of soot is to be avoided, and it should be protected from draughts, since it is difficult to make accurate determinations with a flickering flame. The cause of the discordant results first obtained was found to be wholly due to the condition of the precipitate of barium sulphate, which must be produced with uniform slowness in order to get it uniformly fine. This is best effected by the use of compressed tablets of barium chloride of 1 grm. each. The washings of the bomb, amounting to about 150 c.c., are filtered if necessary, and titrated with $\frac{N}{10}$ sodium carbonate, the acidity giving a rough guide to the selection of a suitable aliquot portion for the sulphur determination. The solution is made up to 200 c.c., and 20 to 50 c.c or more, according to the supposed sulphur content, transferred to the turbidimeter cylinder, which has a mark at 100 c.c. The contents of the cylinder are diluted almost to this mark, shaken, 1 c.c. of hydrochloric acid (1 : 1) is added, water added exactly to the mark, and the mixture again shaken. A tablet of barium chloride is now added, and the tube stoppered and tilted up and down, so that the tablet rolls slowly back and forth through the solution. When precipitation appears to be complete, the remainder of the tablet may be dissolved by rapidly rotating the tube, but violent shaking must be avoided. The turbid liquid is then transferred to a beaker, the candle lighted, and a small quantity of the liquid poured into the tube and the latter put in place. More liquid is then poured down the side of the tube until the image of the flame just disappears. The height, C, of the column of liquid in centimetres is then read, and when this value lies between 2·5 and 17, the amount of sulphur, S, in milligrams in 100 c.c. of the solution under examination is given by the equation: $S = 0{\cdot}6 + \frac{15{\cdot}3}{C}$. When C is less than 2·5 or greater than 17, the value of S may be found from a table given in the paper, or preferably by means of a second experiment, using less or more of the washings of the bomb. The error of the method does not exceed ± 5 per cent. on the sulphur present.

G. C. J.

Quantitative Separation by Means of "Cupferron." O. Baudisch. (*Chem. Zeit.*, 1911, **35**, 913.)—After further experience, the author confirms his previous statement that "cupferron" affords a simple and accurate means of separating iron and copper from other metals (*cf.* ANALYST, 1910, **35**, 78, 453), and gives the following directions for preparing this substance: Sixty grms. of nitrobenzene, 1,000 c.c. of water, and 30 grms. of ammonium chloride, are shaken so as to form an emulsion, and 80 grms. of zinc dust are then added, little by little, while the temperature of the mixture is kept below 18° C. When all the zinc has been added, and the odour of nitrobenzene has disappeared (this takes about thirty minutes), the insoluble zinc hydroxide is removed by filtration, washed a few times with small quantities of water, and the filtrate and washings are cooled with ice to 0° C. The cold solution is now saturated with sodium chloride, when the phenylhydroxylamine separates out in the form of snow-white crystals. The crystals are collected, dried for one hour on filter-paper, then dissolved in 300 to 500 c.c. of ether; the solution is filtered, cooled to 0° C., and a current of dry ammonia gas is passed through it for ten minutes. A slight excess of freshly prepared amyl nitrite is now added; the ammonium salt of nitrosophenylhydroxylamine separates out immediately, and is collected on a filter, washed with ether, dried on blotting-paper, and stored in a stoppered bottle in which a small quantity of solid ammonium carbonate has been placed previously. The compound is quite stable, and its aqueous solution, when rendered ammoniacal, may be kept for months without undergoing decomposition.

W. P. S.

Ferric Sulphate as Standard for Permanganate Solutions. J. Milbauer and O. Quadrat. (*Zeitsch. anal. Chem.*, 1911, **50**, 601-603.)—Thrice crystallised ferrous sulphate is mixed with twenty times its weight of sulphuric acid, and the mixture boiled in a Kjeldahl flask until, when a portion is withdrawn and examined microscopically, it is seen to consist solely of small hexagonal tablets of ferric sulphate. From forty-five to sixty minutes' boiling usually suffices. When cool, the crystals are filtered off in a Gooch crucible in which a porcelain plate has been placed above the asbestos, washed with alcohol and anhydrous ether, and dried to constant weight in a steam oven. The crystals must be preserved in well-stoppered bottles, as they are hygroscopic, but not so hygroscopic as to require special precautions or speed in weighing. For use as a standard, about 1 grm. is accurately weighed out, 25 c.c. of water, 10 c.c. of sulphuric acid, and sufficient zinc, are added, and the mixture warmed moderately until solution is complete. The reduced solution is then titrated against the permanganate. The results agree well with those obtained by the use of the purest oxalic acid or potassium tetroxalate.

G. C. J.

Volumetric Estimation of Ferric Iron by Permanganate after Reduction with Zinc. E. Müller and G. Wegelin. (*Zeitsch. anal. Chem.*, 1911, **50**, 615-623.)—If amalgamated zinc be used for the reduction of ferric salts, it is unnecessary to dissolve the whole of it, as iron is never deposited on amalgamated surfaces. The consumption of amalgamated zinc is much smaller, about 10 per cent. more than is theoretically necessary to reduce the iron, instead of about ten times the theoretical quantity, which is the amount consumed when unamalgamated zinc is used. The use of amalgamated zinc, therefore, reduces the risk of error due to traces of iron in the zinc used. Amalgamation reduces the speed of reaction by about half, but this can be increased by the addition of a small quantity of copper sulphate. With excess of zinc always present, there is no risk of the formation of cuprous salts, which would interfere with the subsequent titration of the iron by permanganate. The decrease in speed due to amalgamation may equally well be counterbalanced by the use of larger zinc surfaces, which, as the zinc does not need to be wholly dissolved, does not greatly increase the amount of zinc passing into solution. If Blair's reductor is to be used—and the authors do not recommend it except when large numbers of determinations have to be made consecutively—the following procedure is recommended: To 100 c.c. of the ferric solution, 5 c.c. of sulphuric acid and 5 to 10 drops of a cold saturated solution of copper sulphate are added. After addition of 3 or 4 sticks of amalgamated zinc (about 5 cm. × 6 mm.), the solution is heated to 100° C. in an atmosphere of carbon dioxide. Reduction should be complete in two hours. In subsequent determinations addition of copper sulphate is, of course, unnecessary.

G. C. J.

Precipitation of Iron with Hydrazine Hydrate. E. Schirm. (*Chem. Zeit.*, 1911, **35**, 897.)—When hydrazine hydrate is added to a solution of a ferric salt, ferric hydroxide is precipitated in a flocculent condition; but on standing, or more rapidly on heating, the precipitate is converted into a fine granular black powder which settles rapidly, and can be filtered and washed very quickly. The precipitation is quantitative. In order to economise the hydrazine hydrate, the solution should first be neutralised with a 10 per cent. solution of ammonia whilst boiling, and, if the ammonia be added in large excess, the precipitation of the ferric hydroxide free from traces of sulphuric acid is insured. A few drops of hydrazine hydrate are then sufficient to convert the precipitate into the black granular form. The dried precipitate is incinerated and strongly heated over the blow-pipe; it is weighed as ferric oxide.

J. F. B.

Separation of Iron and Vanadium by Means of Ether. E. Deiss and H. Leysaht. (*Chem. Zeit.*, 1911, **35**, 869-871 and 878-879.)—The separation of vanadium from iron by Rothe's ether process, as improved by Campagne (*Compt. Rend.*, 1903, **137**, 570), is incomplete even after nine shakings of the ether with acid. With a single shaking, as recommended by Campagne, nearly 10 per cent. of the vanadium may be left in the ethereal layer with the iron, and the error exceeds 1 per cent. after nine shakings. The separation may be made quantitative by the use of a little hydrogen peroxide with the acid used to wash the ether. The solution of vanadium and ferric iron, freed from silica, is evaporated with hydrochloric acid as usual to a syrupy consistency, whereby the vanadium is reduced to the quadrivalent form, in which it is insoluble in ether. The residue is transferred to a separator by the use of the smallest possible quantity (10-15 c.c.) of hydrochloric acid (1 : 1) and the solution cooled. For every grm. of iron present there is next added about 6 c.c. of a mixture made by saturating hydrochloric acid (sp. gr. 1·19) with ether. Such acid takes up about 1·5 vols. of ether with considerable evolution of heat, and it is desired to avoid this evolution of heat in the separator, lest the ferric iron suffer partial reduction by the ether. Ether is added and the mixture shaken, allowed to stand, and separated. The ethereal layer is then shaken with 3 or 4 drops of perhydrol and 10 c.c. of hydrochloric acid (1:1), previously saturated with ether, of which it absorbs 0·3 vol., and the acid aqueous layer is shaken with ether. The acid aqueous layer is then run off, the acid used for washing the ethereal solution containing most of the iron is run into the ether used for washing the first aqueous acid layer, and the principal ethereal solution washed two or three times more with hydrochloric acid (1:1) saturated with ether, the washings being shaken with the ether containing but little iron before adding to the principal aqueous extract. The hydrogen peroxide converts the small residue of vanadium remaining in the ether with the iron after the first separation into pervanadic acid, which, like tetravalent vanadium, is insoluble in ether, but, unlike it, is not liable to become transformed into substances which can pass into the ethereal solution. Vanadium (0·1 grm.) can be separated from 5 grms. of iron with an error not exceeding ± 0·2 mgrm. G. C. J.

Analysis of Monazite Sands. G. Chesneau. (*Compt. Rend.*, 1911, **153**, 429-431.)—In the author's experience decomposition by means of hydrochloric acid is never complete, whilst the use of sulphuric acid always leads to some of the thorium remaining as phosphate in the insoluble residue. The following method works well: The mineral (2·5 grms.) is fused with 6 parts of fusion mixture for an hour, finally at a bright red heat. The product is boiled out with a 1 per cent. solution of caustic soda, which counteracts the tendency of the alkaline carbonate solution to carry some thorium into solution. The insoluble matter is filtered off, washed with cold water, and then treated with hot dilute hydrochloric acid (sp. gr. 1·025). The undissolved matter is filtered off, washed with dilute hydrochloric acid, and submitted to a second fusion, the product of which is treated as before. In this way a hydrochloric acid solution containing the whole of the rare earths, together with some titanium, zirconium, and iron, is obtained; an alkaline solution containing silica and alumina and all the phosphoric acid of the mineral;

and an insoluble residue, consisting of the dioxides of titanium and zirconium, with traces of silica and lime.

The following method for the separation of the rare earths from the hydrochloric acid solution is said to possess advantages over published methods, from which it differs only in details: The solution, freed from heavy metals if present, is evaporated until it contains exactly 4 per cent. of hydrogen chloride—that is, to four-fifths bulk, if the preceding directions have been followed. To the hot solution oxalic acid crystals are added at the rate of 4 grms. for each 100 c.c., and the mixture is allowed to stand for two days. The oxalates of the rare earths are filtered off, washed with oxalic acid solution, dried, and ignited (not above 500° C.). The oxides are dissolved in hot strong nitric acid, and the solution is cleared, if necessary, by addition of a little hydrogen peroxide solution (10 vol.). The solution is evaporated to dryness, and heated at 130° C. until acid fumes are no longer evolved. The residue is taken up in 150 c.c. of water, and the thorium separated by double precipitation with hydrogen peroxide (free from phosphoric acid), a little ammonium nitrate being used to prevent the thorium passing into colloidal solution, which it is especially apt to do in the second precipitation in absence of mineral salts. The filtrates from the thorium are made up to 500 c.c., in 100 c.c. of which the total earths are precipitated by means of ammonia, whilst in another 100 c.c. the cerium is oxidised by means of lead peroxide, and the ceric nitrate titrated with dilute hydrogen peroxide solution. In the remainder of the filtrate from thorium the yttrium earths are separated as double sulphate with potassium sulphate, and lanthanum and the phaseo- and neodidymiums are estimated by difference.

In estimating cerium by the lead peroxide method, a control experiment on an approximately equal amount of cerium should be run simultaneously. To this end a suitable quantity of cerium ammonium nitrate is digested in the cold for one hour with 10 c.c. nitric acid and 2 grms. lead peroxide, stirring from time to time. The filtrate from the thorium is evaporated almost to dryness, and the residue treated in a similar manner. The two liquids are filtered free from lead peroxide by means of asbestos, and are then titrated with hydrogen peroxide solution (0·5 vol.) until the whole of the CeO_2 has been reduced to Ce_2O_3, as shown by the disappearance of the yellow colour.

G. C. J.

Monazite Sand from Travancore, India. (*Bull. Imp. Inst.*, 1911, **9**, 103-105.)—In its natural state this was found to contain over 46 per cent. of monazite, and showed 4 per cent. of thorium dioxide and 28·3 per cent. of cerium, yttrium, and lanthanum oxides on analysis. The monazite readily separated by electro-magnet, and then showed 8·5 per cent. of ThO_2. The Travancore deposit consists, therefore, of material of considerable value in its natural state, and by concentration this can be enhanced beyond that of Brazilian and Carolina monazites (less than 4 per cent. thoria), and resembling that of Ceylon. (*Cf. Bull. Imp. Inst.*, 1905, **3**, 151.)

A. R. T.

Rare-Earth-Containing Sands ("Amang") from Malay States. (*Bull. Imp. Inst.*, 1911, **9**, 99-102.)—The term "amang" is applied to certain heavy sands obtained in the Chinese process of tin-mining in Malaya. The analytical results generally confirm previous findings (*Bulletin*, 1906, **4**, 301), though copper, not previously detected, was present in one sample (2·97 per cent., CuO) from a district where the tin ore concentrates are known to contain the same element. Tungsten was absent in every case, and chromium present in a few cases in traces only. The quantity of monazite in the present samples is uniformly small, and is in no case sufficient to render the "amangs" of value as a source of thoria. The most noticeable feature of three samples is their richness in tinstone (62·35, 67·8, and 76·5 per cent. of the ore). These should be valuable sources of tin, if they are available in quantity.

A. R. T.

Comparison of Methods for Determination of Nickel in Steel. J. J. Boyle. (*Chem. Engineer*, 1911, **14**, 288-289.)—The dimethylglyoxime method is the most accurate available, but the cyanide method is better adapted to routine work in that it occupies only half the time, avoids the use of costly chemicals, and is sufficiently accurate for most routine purposes.

Dimethylglyoxime may be recovered from the nickel salt by mixing this to a paste with water, warming with potassium cyanide, filtering hot and precipitating the oxime at once with acetic acid.

G. C. J.

Method for the Separation of Nickel and Zinc in German Silver and other Alloys. La V. W. Spring. (*J. Ind. Eng. Chem.*, 1911, **3**, 255-256.)—About 0·5 grm. of the alloy is dissolved, the tin being separated by means of nitric acid, the lead by sulphuric acid, the copper electrolytically, and the iron by ammonia. After the addition of 5 grms. of ammonium chloride, the solution is neutralised with hydrochloric acid, then rendered slightly ammoniacal, and the nickel is precipitated by the addition of dimethylglyoxime (*cf.* ANALYST, 1909, **34**, 74). The filtrate from this precipitate is boiled after the addition of hydrochloric acid in order to destroy the excess of dimethylglyoxime, 10 grms. of ammonium sodium phosphate are added, the mixture is neutralised with ammonia, heated, and the zinc phosphate collected and weighed as zinc pyrophosphate.

Estimation of Nitrate and Nitrite Nitrogen in Commercial Calcium Nitrate and Nitrite. Stutzer and Goy. (*Chem. Zeit.*, 1911, **35**, 891.)—The commercial nitrate of calcium obtained as end-product in the oxidation of atmospheric nitrogen invariably contains nitrite, and the nitrite emanating from the same works always contains nitrate. The total nitrogen cannot be estimated accurately by the use of "nitron" after oxidation of the nitrite by hydrogen peroxide. The results are always low. Nor can the nitrate nitrogen be estimated by the use of "nitron" after decomposition of nitrite by means of hydrazine sulphate, as this decomposition is never complete. Nitrite can be estimated by titration with permanganate, and the total nitrogen as nitrate determined by means of "nitron" in the oxidised solution. But the authors prefer to avoid the use of this expensive reagent and determine total nitrogen as ammonia as follows: The nitrate or nitrite (10 grms.) is dissolved in water and the solution diluted to 1,000 c.c. To 50 c.c. of this solution, 50 c.c. water, 50 c.c. caustic soda solution (1 : 2), and 2·5 grms. Devarda metal (Cu : Al : Sn :: 10 : 9 : 1) in powder, are added, and the flask connected to a condenser and receiver containing a measured volume of standard sulphuric acid. When the evolution of hydrogen slackens, the mixture is slowly warmed and the ammonia finally distilled.

G. C. J.

Some Sources of Error in Gasometric Estimation of Nitrate by Schloesing's Method and of Nitrite by Piccini's Method. O. Ruff and E. Gersten. (*Zeitsch. anorg. Chem.*, 1911, **71**, 419-426.)—In presence of arsenious acid or of hydrogen sulphide, Schloesing's method give results which are below the truth. Arsenious acid acts by reducing a portion of the nitric acid to nitrous acid, which passes over as such into the nitrometer and is absorbed by the caustic soda. Hydrogen sulphide causes some of the nitric acid to be reduced to ammonia, which remains in the acid liquid in the flask. The presence of arsenious acid does not affect the accuracy of the results obtained by Piccini's method (*Ber.*, 1881, **14**, 1168) for the estimation of nitrites by reduction with ferrous chloride in neutral solution, but hydrogen sulphide reduces a portion of the nitrous acid to ammonia, and results obtained in presence of soluble sulphides are worthless. G. C. J.

Estimation of Ozone by Means of Alkaline Potassium Iodide Solution. G. Lechner. (*Zeitsch. Electrochem.*, 1911, **17**, 412-414; through *Chem. Zentralbl.*, 1911, II., 261.)—As loss of iodine is liable to take place when ozone is passed through neutral potassium iodine solution, owing to iodine vapour being carried away by the current of gas, the author prefers to pass the gas through potassium iodide solution rendered alkaline with potassium hydroxide. Iodate is formed as the result of the reaction between the ozone and the iodide, and when the solution is acidified the iodine liberated is a measure of the amount of ozone present. The accuracy of the method was tested by estimating the quantity of ozone in an ozoniser by measuring the change in pressure at constant volume; the contents of the ozoniser were then passed through alkaline potassium iodide solution, a current of pure oxygen being employed to drive the gas out of the apparatus, and the iodate formed was titrated as described. Satisfactory results were obtained. W. P. S.

Estimation of Phosphoric Acid by Neumann's Method. I. Bang. (*Biochem. Zeitsch.*, 1911, **32**, 444; through *Chem. Zentralbl.*, 1911, II., 307.)—Instead of expelling the ammonia by heating, the phosphomolybdate precipitate is dissolved in a definite volume of $\frac{N}{2}$ potassium hydroxide solution, and the ammonia is converted into hexamethylenetetramine by the addition of 50 c.c. of neutral formaldehyde solution. The excess of alkali is then titrated. W. P. S.

Colorimetric Estimation of Phosphoric Acid. I. Pouget and D. Chouchak. (*Bull. Soc. Chim.*, 1911 [iv.], **9**, 642-650.)—The reagent employed in the colorimetric method of estimating phosphoric acid described previously by the authors (ANALYST, 1909, 34, 178) should be prepared as follows: Solution 1: Ninety-five grms. of molybdic acid and 30 grms. of anhydrous sodium carbonate are dissolved in 500 c.c. of water; after cooling, 200 c.c. of 36 per cent. nitric acid are added, and the solution is diluted with water to 1 litre. Solution 2: Two grms. of neutral strychnine sulphate are dissolved in about 90 c.c. of warm water, and the solution is then diluted to a volume of 100 c.c. Ten c.c. of solution 1 are mixed with 1 c.c. of solution 2 in order to obtain the reagent. It is not advisable to prepare the reagent from sodium molybdate, as the latter varies in its composition, some commercial samples consisting of neutral sodium molybdate and others of the acid salt. As mentioned previously (*loc. cit.*), the accuracy of the method depends on the proportion of molybdic acid, etc., present in the reagent. W. P. S.

Reduction of Potassium Platinichloride. A. Fiechter. (*Zeitsch. anal. Chem.*, 1911, **50**, 629-632.)—After washing free from sodium platinichloride salt and excess of chlorplatinic acid, the potassium hydrogen platinichloride is dissolved in 50 c.c. of hot water, a spiral of magnesium ribbon is added, and the solution heated to boiling. A few drops of dilute hydrochloric acid are next added, and the boiling continued for two minutes, after which the excess of magnesium is dissolved by addition of a little concentrated hydrochloric acid. The platinum, in fine flakes, quickly settles to the bottom of the beaker, and, if the reduction is complete, the supernatant liquor remains clear and colourless. Even when apparently colourless, it is advisable to add a further fragment of magnesium. If the solution remains clear, reduction is complete, but if more platinum separates, still more magnesium must be added. The reduced platinum is finally filtered off in a Gooch crucible and weighed.

The consumption of magnesium is reduced by keeping the spiral at the bottom of the beaker by pressure with a glass rod. When the quantity of potash to be estimated is 0·05 grm. or less, 0·2 grm. of magnesium usually suffices; with 0·2 grm. potash originally present, 0·5 grm. magnesium may be required to reduce the equivalent amount of platinum; whilst 1 grm. of magnesium will reduce the amount of platinum corresponding to 0·5 grm. potash. G. C. J.

Application of Sodium Paratungstate in the Estimation of Carbon Dioxide in Carbonates and Nitrogen Pentoxide in Nitrates by Loss on Ignition. F. A. Gooch and S. B. Kuzirian. (*Zeitsch. anorg. Chem.*, 1911, **71**, 323-327.)—Sodium paratungstate serves as well as any of the salts which have been recommended for expelling carbon dioxide and nitrogen pentoxide from carbonates and nitrates on fusion. It is more easily prepared than anhydrous borax, silica, or sodium metaphosphate, and gives rise in use to less viscous substances than do borax and silica, and fusions with it can therefore be effected at lower temperatures. The authors prefer it also to potassium bichromate, which may lose oxygen at the temperatures necessary to decompose carbonates.

Sodium paratungstate, which corresponds approximately to the formula $5Na_2O.12WO_3$, is prepared by dehydrating and fusing sodium tungstate, adding an equal weight of tungsten trioxide, previously ignited to expel ammonia and insure complete oxidation, and continuing to heat the mixture until a clear fusion results. The cooled product, which is readily powdered, is preserved in a desiccator over sulphuric acid, though it is not particularly hygroscopic. It contains approximately half its weight of tungsten trioxide, and 1 grm. will therefore displace somewhat more than 0·1 grm. of carbon dioxide or 0·25 grm. of nitrogen pentoxide. In use, the mixture of the paratungstate with carbonate or nitrate is heated at first at a very low temperature, and then kept in a state of fusion for five minutes. After weighing, the fusion is repeated, but there is seldom any measurable loss on reheating.

G. C. J.

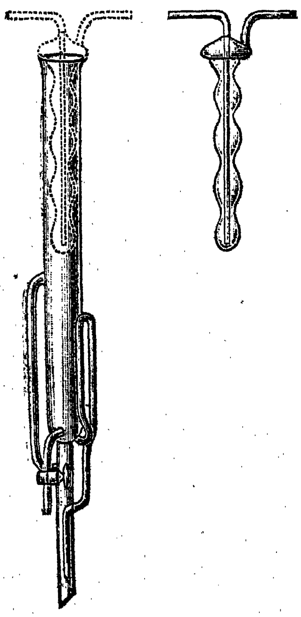
Estimation of Tin in Ores. R. J. Morgan. (*Chem. Engineer*, 1911, **14**, 289-291.)—Stick soda (6 to 8 grms.) is heated in a covered iron crucible to expel moisture, and, when it has cooled sufficiently to be once more solid, 5 grms. of the powdered ore are added, and the contents of the covered crucible heated gently at first, and afterwards to dull redness until fusion is complete. The melt is boiled out with as little water as possible, and the crucible and lid rinsed with a little dilute hydrochloric acid, which is added to the alkaline solution. Strong hydrochloric acid is added cautiously so long as there is effervescence, and when this ceases a further 20 c.c. is added and the solution heated to boiling-point and transferred to a flask. There should be nothing undissolved save, possibly, a little gelatinous silica. Three shoeing nails are bent up and pushed into the flask, the contents of which are boiled for fifteen minutes and then filtered through glass wool, the latter being washed with 50 c.c. of water. Shoeing nails being very soft, pure iron, remove copper and arsenic without risk of introducing other impurities. In the filtrate the tin is estimated by Low's method (reduction to stannous condition by metallic antimony, followed by rapid cooling in atmosphere of carbon dioxide and titration with iodine).

G. C. J.

Sampling of Coal in the Mine. J. A. Holmes. (*U.S. Bureau of Mines*, 1911, *Technical Paper I.*, pp. 16.)—The purpose of this paper is to describe the methods now followed by the Bureau of Mines and U.S. Geological Survey in an endeavour to take mine samples that fairly represent the beds of coal that are examined, and that show the commercial possibilities of the beds. It describes and illustrates the sampling kit issued by the Bureau to their collectors, as well as a simpler and lighter outfit for the use of field men who work at long distances from a base of supplies. The sampler is advised first to study a map of the mine, next to examine the mine itself, and then, having determined the number of samples to be collected, to select suitable places for taking these. The number of samples should not be less than four from any mine, nor less than one for each 200 tons of coal mined per day, and should be greater from mines in which the quality of the coal varies greatly. Detailed directions are given for cleaning off the face of the coal, cutting the sample, breaking it down and quartering it. Judgment and experience are demanded of the sampler in deciding what to include in the sample. Usually partings more than ⅜ inch thick and lenses or concretions of "sulphur" or other impurities more than 2 inches in maximum diameter and ½ inch thick are excluded, but, if in the judgment of the sampler, they are not being excluded by the miner or at the bank, they should be included. The collectors of the Bureau are required to fill in the answers to some 300 questions concerning each mine, and copies of the forms used are appended to the paper.

G. C. J.

APPARATUS, ETC.



Improved Soxhlet Condenser. O. Silberrad. (*Chem. News*, 1911, **104**, 54.)—This modified form o combined condenser and extraction apparatus avoids the unsatisfactory cork joint between the tube and condenser. A bulbous Walters condenser simply fits on to the (elongated) tube of the Soxhlet apparatus.

A. R. T.

New Type of Automatic Inflammable Gas and Vapour Detector. A. Philip and L. J. Steele. (*J. Soc. Chem. Ind.*, 1911, **30**, 867-872.)—The detector described and illustrated in the paper depends, like that of Liveing, on the catalytic action of platinum, but means are provided for preventing the platinum coils from becoming overheated, and thus fusing or having their capacity as catalyst, impaired. In its stationary form, the detector may be fixed anywhere, and air is continually drawn by means of a pump from the space (ship's bunkers, etc.) the atmosphere of which needs to be kept under observation, and is forced through the apparatus. At a certain concentration of inflammable vapour, attention is called to the fact by the ringing of a bell and the lighting up of a red lamp and the extinguishing of a white one. At the same time, the stream of air containing the combustible gas is cut off, thus preventing overheating of the platinum coils. The apparatus can be adjusted to signal when the proportion of combustible gas is one-third or any other fraction of the explosive limit. In its ordinary form, the apparatus is wholly enclosed in a massive casing which cannot be tampered with. A portable form is also described, in which not only the heavy casing but the electric motor-pump is dispensed with, the latter being replaced by a hand-pump.

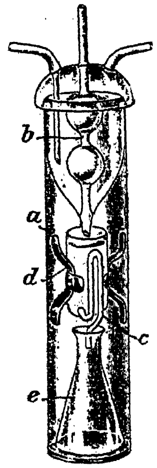
G. C. J.

Methods of Testing Safety Lamps or Other Apparatus for the Detection of Combustible Gases and Vapour. A. Philip and T. Stenhouse. (*J. Soc. Chem. Ind.*, 1911, **30**, 861-867.)—The paper describes the methods available for testing apparatus designed for the detection of inflammable vapours. The sources of combustible gas for experimental purposes are enumerated, and methods described for the mixture of the combustible gas with air, so as to obtain an atmosphere suitable for such tests as may need to be carried out. Methods are also described for determining the chemical composition of mixtures of air and inflammable vapour prepared by the methods described in the earlier part of the paper. The paper is accompanied by diagrams showing convenient arrangements of apparatus.

G. C. J.

A New Extraction Apparatus. G. A. Quincke. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 171-172.)—The apparatus shown in the illustration consists of a Jena-glass vessel, *a*, which is heated by means of a water-, sand-, or oil-bath; the vapours of the solvent contained in the flask *c* pass into the condenser *b*, which fits into the top of the glass vessel, and the drops of condensed solvent fall on to the material to be extracted, contained in the siphon vessel *e*. The latter vessel is supported at a convenient height by means of the springs *d*. Corks or rubber stoppers are dispensed with in the apparatus.

W. P. S.



Flask for Distillation of Tar. C. E. Brown and G. B. Shadinger. (*J. Ind. and Eng. Chem.*, 1911, **3**, 616.)—In order to avoid the "bumping" of wood-tar during distillation, owing to the presence of water, the authors employ an ordinary distilling-flask with side-tube and short neck, and having a thin sheet of asbestos paper wrapped once round the neck above and below the side-tube. This asbestos covering has wound over it a few turns of No. 30 Nichrome resistance wire, and a thick covering of asbestos is placed over all and securely fastened. A current of 15 to 30 volts passed through the wire furnishes heat sufficient to keep the neck of the flask at the required temperature, and to prevent condensation at this point.

A. R. T.

Apparatus for Electrolytic Estimations with Mercury Cathode. P. Baumann. (*Chem. Zeit.*, 1911, **35**, 854-855.)—The cathode (Fig. 1) consists of a flat-bottomed cylindrical vessel of Jena glass, 40 mm. wide, 70 mm. high, and 1 mm. in thickness of wall, and weighs less than 25 grms. without the mercury, of which about 60 grms. is used. A platinum wire, 0·8 mm. thick, is fused through the glass near the bottom, and penetrates 1 cm. into the mercury. The other end of the wire is bent as shown, and its point sealed to the glass about 5 mm. above the level of the mercury. The tubulus, shown on the left, is about 4 mm. above the bottom of the vessel, 6 to 7 mm. in diameter, and carries, by means of a ground-joint, the bend with bulb and glass stopcock. The inner end of the bend is again bent at right angles, and cut off short so as just not to dip into the mercury. The internal diameter of the bend is 2·5 mm., and that of the bulb 11 mm. The bulb serves to catch any mercury which may be carried over in drawing off the electrolyte or water used to wash the cathode. Before introducing the electrolyte the bend is thoroughly dried internally, to prevent the diffusion over its surface, during electrolysis, of a film of the electrolyte, which would thus escape the action of the current. When, at the end of the electrolysis, the electrolyte has been withdrawn and the cathode washed with water, the bend is replaced by a ground stopper, and the washing with alcohol and ether conducted with the help of a siphon, the cathode vessel being removed from its stand and inclined, to reduce the surface of the mercury to be washed. The apparatus is made by Schott und Gen., Jena.

An anode suitable for use with the above, and made by W. C. Heraeus, Hanau, is described in the paper.

The electrical heater (Fig. 2), also intended for use with the above, consists essentially of the platinum-wire helix, shown at the bottom of the figure, and the unshaded part of the glass tubular structure, which is filled with kerosin. The current enters and leaves the apparatus at the binding screws *a*, *a'*, which are connected to stout copper wires, 3 mm. in diameter, which pass along the horizontal glass tubes (4·5 cm. long) and down the tubes *b*, *b'*, which are 8·5 cm. by 6 mm., and sealed at *c*, *c'*. The copper wires are connected to short, stout pieces of platinum wire, 1 cm. by 1 mm., which are fused through the glass at *c*, *c'*, and connected to either end of the heating coil, which has a resistance of 0·5 ohm. The tube *d*, which serves to support the apparatus in the clamp shown, and its two branches, which contain the coil, are filled with kerosin, and the tubes *b*, *b'* are filled with mercury to above the level of the horizontal tubes. The tubes *b*, *b'* and *d* are all sealed with wax or corked. A current of 10 to 11 ampères will heat 30 c.c. of liquid to 70° C. in four or five minutes, and 6 ampères will maintain this temperature. From centre to centre *b* and *b'* are 38 mm. apart, and 28 mm. from *d*. The apparatus is made by R. Goetze, Leipzig.

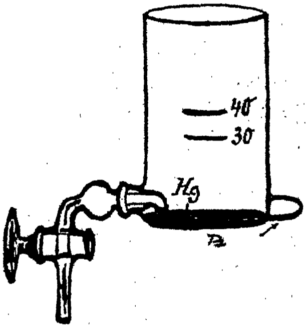


Fig. 1.

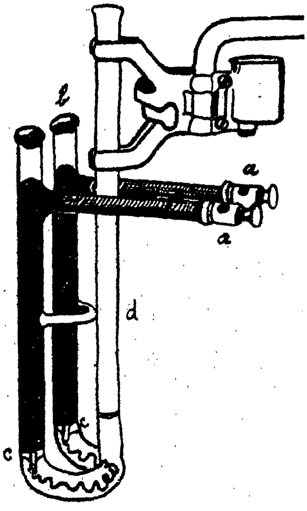


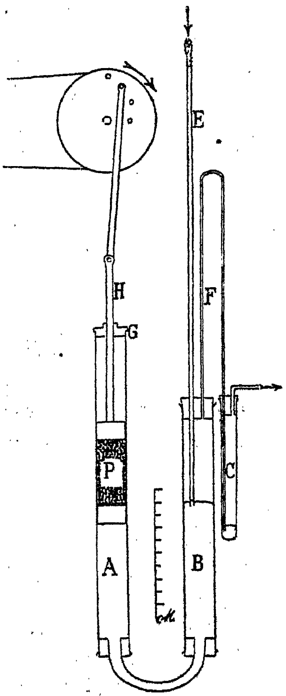
Fig. 2.

G. C. J.

Apparatus and Methods for Sampling and Analysis of Furnace Gases. J. C. W. Frazer and E. J. Hoffman. (*U.S. Bureau of Mines, Bulletin* 12, p. 20.)—The apparatus and methods described in this Bulletin were devised by the authors in connection with their investigations on the improvement of furnace conditions and on efficiency in the use of fuel. Their gas-sampling tube is no advance on that of Huntly (ANALYST, 1910, **35**, 285), but their apparatus for instantaneous sampling is both novel and useful. Frequently it is desirable to ascertain the composition of gases at some point in a furnace at a certain definite instant, and getting an instantaneous sample from a space at a high temperature is attended with difficulty. There must be no dead space between the sampling tube and the point of collection of the sample; in other words, the sampling tube must itself be introduced into the furnace. The apparatus figured by the authors is adapted to this purpose, and is, of course, contained in a steel water-jacket. The sampling tube is exhausted, fixed in its jacket, and the whole apparatus introduced into the furnace, when the sampling tube is placed in connection with the point from which a sample is wanted by simple pressure on a knob. Pulling the knob closes the stopcock of the sampling tube again, and the apparatus is withdrawn. As it need not be in the furnace more than thirty seconds, and as the quantity of water in the jacket is considerable, it is unnecessary to provide for a circulation of water. The apparatus is somewhat complicated, but, having regard to what it accomplishes, not unreasonably complicated. It is fully illustrated and described in the Bulletin, which may be obtained free from the Director of the Bureau of Mines, Washington.

The Bulletin also illustrates and describes an apparatus for exact gas analysis, for which no claim to originality is made, its essential features—the automatic compensator of Pettersson, and Bleier's burette with two limbs, one of them bulbed—being familiar; but it appears to possess some advantages over other forms of apparatus. Experiments are described showing that 100 c.c. of gas can be measured accurately to 0·01 c.c. With volumes less than 50 c.c. the readings can be more precise. Finally, there is a description of a novel device for calibrating burettes and other measuring apparatus, by means of which some fifty observations can be made in an hour with a probable error of less than 0·01 c.c. G. C. J.

Detection of Gas in Sealed Tube Reactions. W. H. Warren. (*J. Amer. Chem. Soc.*, 1911, **33**, 1417-1418.)—When one of the products of a sealed tube reaction is a gas, this cannot be examined if an ordinary Carius tube is used, as it escapes when the tip of the tube is heated in the flame. If a suitable stopcock be sealed to the end of the Carius tube after the introduction of the reagents, any gas formed during subsequent heating may be drawn off from the cooled tube to any appropriate apparatus for identification or analysis. The stopcock must, of course, be made of similar glass to that used in the construction of the tube, and the following dimensions have been found suitable: Length of plug of stopcock 2·7 cm., taper 8 to 10 mm., bore 1·5 mm. On the side remote from the sealed tube, the cock carries a tube of any convenient dimensions for connecting with gas apparatus, that figured in the paper being 4 cm. long by 4 mm. internal diameter. From the other side of the cock springs another tube which quickly widens to 1·3 cm. internal diameter and is 2 mm. thick in the wall. This tube, which is conveniently about 7 cm. long, is sealed to the Carius tube after the latter has, if necessary, been drawn out slightly to be of the same diameter. If the joint is well annealed, accidents are not more common than with ordinary sealed tubes, and, if the stopcock is wrapped in cotton wool, it can often be saved even if the tube breaks. When the experiment is concluded and the gas drawn off, the stopcock is cut off and used again. The best lubricant for the plug is graphite. Stopcocks of the pattern described in "R" glass are made by C. Desaga, Heidelberg. G. C. J.



Simple Laboratory Suction Pump. F. Liebert. (*Chem. Weekblad*, 1911, 8, 382-383; through *Chem. Zentralbl.*, 1911, II., 329.)—In the pump here shown, the small glass receptable P is loaded with shot and has a stem H passing through the top of G. As it rises or falls in the tube A, which is charged with mercury, it causes the mercury in the tube B to fall or rise correspondingly. With the fall of the mercury in B, gas is drawn from E, whilst a rise in the mercury impels the gas through F into the test-tube C, which also contains mercury. As the mercury sinks in B, there is a rise of the mercury from C into F, which forms a sufficient seal during the suction process. On the other hand, when pressure is applied, the tube E is sealed by the rising mercury. C. A. M.

REPORTS, ETC.

Report on the Work of the Laboratories, Survey Department, Egypt (1910). A. Lucas. The work of this laboratory includes the chemical analysis of miscellaneous articles for composition, purity, etc., the physical testing of cements, bricks, etc., and the photometrical examination of illuminating gas and incandescent mantles. A total of 4,330 samples was examined during the year, 1,703 of these samples being analysed chemically, including sixty-five samples of foods. Eighteen samples of butter-fat analysed were found to be of normal composition. W. P. S.

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REVIEWS.

RUBBER. By PHILIP SCHIDROWITZ. London: Methuen and Co., Ltd., 1911. Price 10s. 6d. net.

Owing to a number of causes rubber has attracted an unusual amount of attention during the past few years, and much useful work, both from the scientific and from the more purely technical sides, has been devoted to the solution of some of the more important problems connected with its production and employment. For many years prejudice and rule-of-thumb reigned supreme, and it was widely, if not universally, assumed that the various commercial rubbers had their fixed positions in the scale of quality, and that their properties—good, bad, or indifferent—were inherent and beyond the control of the technologist. Not the least useful outcome of recent work has been the recognition of the fact that this is only true to a certain extent, and that by the application of better methods much may be done to improve the inferior qualities, and so materially to lessen the distance between the best and the worst. Perhaps the most valuable portions of the work under review, and those which afford the greatest justification for its appearance, are the descriptions and criticisms of the various investigations which have, directly or indirectly, been instrumental in bringing about this improved state of affairs. The author has, himself, done good work in this fascinating and important field, and this, coupled with his first-hand knowledge of the subject in its various aspects, entitles him to claim for his views—even when, as is sometimes the case, these are decidedly speculative—the serious attention and respect of his readers. The work necessarily covers much of the ground occupied by previous authors, such as Weber, Clouth, Wright, Seeligmann, and others; but it is written in the light of the results of modern researches, and a great deal that is new and suggestive will be found even in those portions of the book where it might appear to be difficult to say much that has not already been said. The chapters dealing with wild rubber and the plantation industry are excellent, and if the same cannot be said of that referring to the production of so-called "industrial rubbers," it is clearly because the author has not felt himself at liberty to say what he knows. This is a matter for regret, since it detracts a little from the value of the book as a general survey of the whole industry. The methods adopted and the solvents employed in the winning of these rubbers are such very open secrets that probably no harm would have been done to anyone by a somewhat fuller reference to the subject. Seeing that such immense and increasing quantities of these rubbers are finding their way on to the market, it seems a pity that the whole subject should have been dismissed in the space of eight pages. A similar criticism applies to the discussion of "reclaimed" rubber.

The chapter dealing with the chemistry of crude rubber is clearly and concisely written, and in dealing with a subject where such magnificent superstructures have often been reared on such slender foundations of fact, the author cannot be blamed if he has erred on the side of condensation and exclusion. The view that the resins usually associated with the various crude rubbers are not genetically related to the rubber hydrocarbons is probably correct; but, nevertheless, they are of importance from several points of view, and their treatment is, in the opinion of the writer, scarcely adequate. The chapter on the chemical analysis of crude and vulcanised rubber is rather disappointing, in the sense that it contains little that is novel, either in the description of new methods or in the critical discussion of old ones. The prefatory remarks of the author, however, disarm criticism on this point, and it only remains for the reviewer to refer to the fact in passing. The author is, of course, right in stating that "a commercial valuation cannot be undertaken on the basis of chemical analysis alone," and that the chemical results should be supplemented by physical tests and by vulcanisation experiments. Still, one cannot help feeling that undue emphasis has been laid on the inadequacy of chemical results and on the difficulties attending the evaluation of commercial rubbers. Very large quantities of rubber are bought and sold daily on the results of simple chemical analysis, aided, of course, by the expert knowledge of the buyer, who in any case is in a better position to know what class of rubber he requires than is the chemist, no matter how expert he may be. Having said so much by way of criticism, there remains nothing but praise for the work, which is clearly written, and provides on the whole an excellent survey of the present condition of the rubber industry. The paper and printing are good, and the illustrations are excellent. There are very few typographical errors, but with a view to their correction in future editions the following, which have caught the reviewer's eye, may be noticed: "Diamethyl" for dimethyl, on p. 119; "hydriod" for hydriodic, on p. 120; "phenonema" for phenomena, on p. 124; and "adsorption" for absorption, on p. 249. "Ido-bromine," on p. 151 is, moreover, not a very intelligible expression. A. CHASTON CHAPMAN.

THE CHEMISTRY OF ENZYMES (ALLGEMEINE CHEMIE DER ENZYME). By HANS EULER. Wiesbaden: J. F. Bergmann, 1910. Pp. viii, 238. Price 5·70 m.

In this book the author aims at giving a succinct account of enzymes and enzyme action, the various chapters being devoted to the descriptive chemistry of enzymes, their physical properties, activators and inhibitants, the chemical dynamics and statics of enzyme action, enzymic synthesis, and the specific character of enzymes. This comprehensive scheme necessitates a condensed mode of treatment, but the work, in spite of this, is readable and not too greatly loaded with details. The author takes a broad view of his subject, and admits that enzymes in many cases may do more than merely accelerate or diminish the rate of a reaction; that they may not only combine with the substrate and the products formed from it, but may take part in and affect the condition of equilibrium. An examination of the numerous data quoted concerning the course of enzymic reactions shows that the due recognition of this possibility and a careful study of the various factors involved in each particular case of enzyme action are absolutely necessary if any great progress is to be made in the elucidation of the nature and mode of action of these mysterious agents.

As regards the actual chemical character of enzymes, no direct information is as yet available; in spite of many attempts at purification, no organic substance has been isolated about which it can be definitely said that it is a chemical individual and has the properties of one of the natural enzymes.

The work concludes with a short appendix containing a brief description of some of the most important methods of obtaining enzyme preparations and ascertaining their activity.

The author has undoubtedly succeeded in presenting in comparatively small space a general view of our knowledge of enzymes and enzyme action. Greater detail might appear to be desirable in several cases, notably in the discussion of the important subject of enzymic synthesis, but on the whole the relative importance of the various branches of the subject is adequately realised. The reader interested in any particular section of the subject will find here a general statement bearing on the point, and will be guided by the copious references to the literature. More than this it would be unreasonable to demand. A. HARDEN.

NOVEMBER, 1911. Vol. XXXVI., No. 428.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE monthly meeting of the Society was held on Wednesday evening, November 1, in the Chemical Society's Rooms, Burlington House. In the absence of the President, the chair was occupied by Dr. Bernard Dyer, F.I.C., Past-President.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. R. D. Carty, W. K. Walton, and G. F. Wesley Martin, were read for the second time; and certificates in favour of Messrs. Shelton Gottlieb Agar, "La Fontaine," Clifton, Guernsey, Assistant Public Analyst for Guernsey; George Stephen Barton, 8, Exhibition Road, South Kensington, S.W., analyst and bacteriologist to the Belgrave Dairy Co., Ltd.; Stanley W. Bridge, A.I.C., "Dunsmore," Park Hill Road, Croydon, assistant to Mr. E. Hinks, B.Sc., F.I.C.; Richard Victor Briggs, 4, Pollock Street (P.B. 279), Calcutta, analytical and consulting chemist; Francis Archibald Mason, 26a, Great George Street, Leeds, analytical and consulting chemist; William Bristow Saville, 117, Vassall Road, Brixton, S.W., assistant to Messrs. Harland and Brown; George Alfred Stokes, 191, Edgware Road, Paddington, W., assistant to Mr. A. W. Stokes, F.I.C.; and Francis Clifford Dyche-Teague, B.Sc. (Birmingham), A.I.C., 28, King Street, Portman Square, W., analytical and consulting chemist, were read for the first time.

Mr. H. C. Huish was elected a member of the Society.

The following papers were read: "Notes on Shrewsbury and Knapp's Process for Estimating Cocoanut Oil," by H. S. Shrewsbury, F.I.C., and Arthur W. Knapp, B.Sc., F.I.C.; "Note on the Examination of Finnish Turpentine," by L. M. Nash, F.I.C.; "Note on a Counterfeit Gold Coin," by H. S. Shrewsbury, F.I.C.; "Note on the approximate Estimation of Starch by Iodine," by L. Reed, F.I.C.; "Note on the Gravimetric Estimation of Phosphorus in Milk," by E. H. Miller; 'Kobert's Reagent as a Test for Salicylic Acid," by J. McCrae, Ph.D., F.I.C.; and "Precipitation of Nickel Compounds and Preparation of Spongy Nickel," by W. H. Low.

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INTERNATIONAL CONFERENCE ON FOOD ANALYSIS.

As the result of some correspondence which they had had with the Foreign Office, the Local Government Board in June, 1910, addressed a letter to the President, suggesting that the Society should send a representative to the International Conference which was to be held in Paris on June 27, and succeeding days. This Conference was convened by the French Government for the purpose of discussing two main questions; first, the means to be adopted for the unification of methods of expressing the analytical results obtained in the analysis of food products; and, secondly, that of the unification of the analytical methods themselves. Mr. A. Chaston Chapman was appointed to act as the Society's representative, the other British delegates being Dr. G. S. Buchanan and Dr. G. W. Monier-Williams, representing the Local Government Board; Dr. J. J. Dobbie, Director of the Government Laboratories; Mr. E. J. Connah, Chief Chemist of the Customs Laboratory; and Dr. Nestor Tirard, Secretary of the Pharmacopœia Committee of the General Medical Council. The following countries were also represented: Argentina, Belgium, Bulgaria, China, Denmark, France, Greece, Holland, Hungary, Italy, Mexico, Norway, Portugal, Sweden, Switzerland and Uruguay, and the Meetings were held under the Presidency of Dr. Bordas, the Principal of the Laboratories of the French Ministry of Finance.

The following is a translation of the result of the deliberations of the Conference, and of the rules which have been proposed for the expression of analytical results obtained in the analysis of food products. To these recommendations all the British delegates signified their adhesion.

I. The members of the International Conference which was held in Paris on June 27, 1910, are in agreement in recognising that the unification of the methods of analysis of food products involves, in the first place, the unification of the methods of expressing the analytical results obtained conformably with the rules laid down hereafter.

II. The Conference expresses a desire that the French Government should take the initiative in considering a project for the creation of a permanent International Bureau, with laboratories attached.

The object of such laboratories would be to study the methods of analysing food products with a view to their possible unification, as well as the preparation of such tables of comparison (*concordance*) as might be considered necessary.

The Conference further expresses the hope that this project may be submitted by the French Government as soon as possible to all the countries represented at the Paris Conference.

III. The delegates agree to recommend to the recognised authorities of their respective countries the adoption of the methods suggested by the Conference. They express the wish that their decisions may be sanctioned by an International Convention, and, in particular, that measures shall be taken in the countries represented to bring into general use the manner of recording analytical results as adopted by the Conference.

RULES FOR THE UNIFICATION OF THE EXPRESSION OF ANALYTICAL RESULTS.

1. *Notation.*—The methods of notation are to be those adopted by the International Committee of Weights and Measures. The atomic weights employed are to be those established by the International Commission on Atomic Weights.

2. *Mass (Quantity of Matter).*—The practical unit of weight is the gram—the one-thousandth part of the international kilogram. This is in conformity with the definition of the General International Conferences on Weights and Measures, and introduced into the laws of those countries which have signified their adhesion to the Metric Convention.

3. For products the quantity of which is estimated by weight (*dont on évalue la quantité par des pesées*), the results of the analysis giving their composition are to be expressed in grams or milligrams per 100 grms. of the product. These results are indicated as follows:

g. %g. or mg. %g.
g/100 g. or mg/100 g.
g. p. cent. g. or mg. p. cent. g.

4. *Volume.*—The unit of volume is the litre—the volume of a kilogram of pure water under the conditions defined by the General International Conferences on Weights and Measures. The practical unit of volume measurement is the cubic centimetre, equal to the one-thousandth part of the litre.

5. The litre is represented at the temperature of t° by the volume of g grams of distilled water weighed in air with brass weights. A table giving the values for g for various temperatures is appended.

6. In the case of substances that are measured by volume, the results of the analysis are to be stated in grams or milligrams per litre of the substance. Thus—

g/L. or mg/L.

The results can at the same time be expressed in any other desired manner.

7. *Temperature.*—Temperatures are to be given in terms of the normal scale adopted by the General International Conferences on Weights and Measures—that is, the Centigrade scale of the hydrogen thermometer, which has for its fixed points—

(1) The temperature of melting ice (0°).
(2) That of the vapour of distilled water boiling under normal atmospheric pressure (100°).

As far as possible, the boiling-points should be subjected to the usual corrections. They are in that case to be followed by the letters "corr."

8. *Calorimetric Measurements.*—Thermo-chemical results must be expressed in large calories with the letters "gr. cal." (the quantity of heat required to raise the temperature of 1,000 grms. of water 1° Centigrade).

9. *Pressure.*—Pressures must be expressed in millimetres of mercury at 0° and under normal conditions of gravity.

10. *Density.*—Density is the ratio of the mass of a given volume of a body to the mass of the same volume of distilled water at 4° and under normal pressure.

11. Since the majority of existing tables contain the values at 15° compared with water at 15°, densities are in practice referred to those conditions (alcoholic solutions, and especially solutions of various acids, oils and essences).

An exception is made in the case of solid fats, but the temperature *T* at which their density has been taken, and also the temperature *t* of the water to which the density is referred, are to be stated as follows:

100°/15°, 40°/40°, etc.

12. Densities are not to be given in arbitrary units (Baumé, Tessa, Cartier, etc.).

13. The alcohol content of alcoholic liquids must be given in grams of alcohol, either per litre or per 100 c.c., and at the same time in volumes of alcohol according to the method in vogue in the particular country, but preferably in volumes of absolute alcohol contained in 100 volumes of the liquid analysed.

14. *Refractive Index.*—Refractometric measurements are to be expressed as refractive indices with reference to air, for the spectrum line D, and at a temperature of 25°, but at a temperature of 40° for fats.

When, however, it is impossible to work at the temperatures of 25° or 40° as mentioned above, the refractive index may be taken at another temperature *T*, but this must be designated by the expression "refractive index (*T*)."

15. *Polarimetric Results.*—Polarimetric readings are to be expressed in terms of degrees of arc, with centesimal fractions, for a tube of 20 cm., at a temperature of 20°, and for yellow light (D).

In the case of solids, the nature of the solvent and the concentration of the solution should be indicated.

16. *Acidity.*—Whatever the nature of the acid (whether fixed or volatile, free or partly combined), the acidity is to be expressed by the number of cubic centimetres of normal, decinormal, or centinormal solution corresponding to 100 grms. of substance or to 1 litre of liquid, the following notation being employed:

$$\text{cm}^3\text{N.},\quad \text{cm}^3 1/10\text{N.},\quad \text{cm}^3 1/100\text{N.}$$

In the case of butter and other fats, the acidity is to be referred to 100 grms. of the actual fatty matter.

Side by side with the above, results may be given in terms of acetic, tartaric, malic acid, etc., according to the nature of the product, or arbitrarily in terms of sulphuric acid, or in any other way.

In addition, the method employed and the indicator used should be mentioned whenever these have a bearing upon the results.

17. The saponification value is to be given in cubic centimetres of normal solution corresponding to 100 grms. of the fat analysed. It may be accompanied by the Köttstorfer number.

18. *Alkalinity.*—Whatever the nature of the bases, the alkalinity is to be expressed by the number of cubic centimetres of normal, decinormal, or centinormal

solution corresponding to 100 grams or to 1 litre of the substance analysed, the following notation being used:

$$\text{cm}^3\text{N.},\quad \text{cm}^3 1/10\text{N.},\quad \text{cm}^3 1/100\text{N.}$$

The alkalinity may at the same time be expressed in any other manner. The indicator used should also be stated when this has any bearing on the results obtained.

19. The alkalinity of the ash of a substance, expressed in the manner stated above, should be referred to 100 grms. or to 1 litre of the substance.

20. *Reducing Sugars.*—Reducing sugars the nature of which is not stated are to be expressed in grams of glucose per 100 grms. or per 1 litre of the substance analysed.

21. *Iodine Value, Bromine Value, etc.*—The iodine and bromine values represent the number of grams of halogen, calculated as iodine or bromine respectively, which combine with 100 grms. of the substance.

In the case of butter and other fats, the results are to be referred to 100 grms. of the actual fat.

22. *Proteins.*—When any factor other than 6·25 is used in calculating proteins from the amount of nitrogen, this factor should be given in parentheses.

23. *Analysis of Spirits (Special Regulation).*—The ethers are expressed in terms of acetic ether, the aldehydes as acetaldehyde, the higher alcohols as isobutylic alcohol or amylic alcohol, stating which of the two is taken. The volatile acids are to be expressed in terms of acetic acid.

These constituents are to be expressed in milligrams per litre of the liquid, and at the same time in milligrams per 100 c.c. of absolute alcohol contained in the liquid analysed.

The extractive matters and the fixed acidity (calculated as acetic acid) are to be expressed in grams per litre of the liquid analysed.

The letters C.I. may be used to indicate that the analytical results are expressed in conformity with the preceding rules.

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THE PRECIPITATION OF NICKEL COMPOUNDS AND PREPARATION OF SPONGY NICKEL.

By WILSON H. LOW.

(*Read at the Meeting, November* 1, 1911.)

Textbooks on both qualitative and quantitative analysis are somewhat limited in their remarks concerning the behaviour of nickel solutions with ammonia, and there is little in them to suggest that ammonia is a possible reagent for the precipitation of nickel salts. In Roscoe and Schorlemmer's "Chemistry," however, the statement is made that nickel is precipitated as hydroxide by the fixed alkalis, that the precipitate dissolves in ammonia, *and separates out as a green crystalline powder on boiling the ammoniacal solution.*

This statement was found to be correct. The nickel separates in a form easy to filter and wash, and of course is free from fixed alkali. This would be an ideal method for determining nickel were it not for the fact that the precipitation is not complete, but the filtrate contains enough nickel in solution to be slightly greenish in colour.

Further addition of ammonia and boiling failed to produce more precipitate. With the hope that hydrazine might give better results, the original nickel solution was boiled with an excess of hydrazine hydrate. This produced a precipitate from neutral solutions of varying colour, but with an excess of the reagent the precipitate was a delicate blue-violet. This precipitate was crystalline, could be easily filtered and washed, and, except in colour, appeared physically much the same as the precipitate produced by ammonia. But here again it was found that the filtrate contained nickel, which further addition of the reagent and boiling failed to precipitate.

While neither ammonia or hydrazine hydrate gave filtrates free from nickel, it was found that if the filtrate from the ammonia precipitation was treated with hydrazine hydrate the remaining nickel was completely precipitated, as evidenced by the filtrate being clear and colourless. Operating in the opposite manner, and adding ammonia to a hydrazine hydrate filtrate, no further precipitate was formed. The explanation appeared obvious: Hydrazine will precipitate nickel completely from solutions containing ammonium salts, but ammonia will not cause a complete precipitation in solutions containing hydrazine compounds. This proved to be the case, and, starting with a nickel solution containing ammonium chloride, the hydrazine precipitated the nickel completely.

Either the precipitate produced by ammonia or hydrazine may be easily reduced to spongy nickel by heating in hydrogen. The spongy nickel is free from fixed alkali.

LABORATORY OF THE CUDAHY PACKING COMPANY,
SOUTH OMAHA, NEBRASKA, U.S.A.

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KOBERT'S REAGENT AS A TEST FOR SALICYLIC ACID.

By J. McCRAE, Ph.D., F.I.C.

(*Read at the Meeting, November* 1, 1911.)

KOBERT'S reagent (three drops of formaldehyde solution in 3 c.c. of sulphuric acid), so useful in the detection of morphine, gives a characteristic rose colour with salicylic acid (or salicylates).

When Kobert's reagent is added to dry salicylic acid, the colour develops fairly quickly, but the colour does not appear quite immediately. The reaction is most clearly seen when about $\frac{1}{50}$ mgrm. of salicylic acid is dissolved in two drops of sulphuric acid, and a drop of Kobert's reagent then added; the fine rose colour develops within a few minutes.

The same result is obtained with acetyl-salicylic acid (aspirin and xaxa), and also with salol; in the case of salol the colour develops more quickly.

Sulphuric acid gives a red colour with salicine, but the colour obtained with Kobert's reagent is deeper and richer. This colour reaction can be best seen by dissolving about $\frac{1}{50}$ mgrm. of salicin in three drops of sulphuric acid, and to this red solution adding one drop of Kobert's reagent in such a way that mixing does not take place; a deep red colour is produced.

Kobert's reagent gives the following characteristic colours with other substances: Phenol, red-violet; catechol, violet (redder than the morphine colour); resorcinol, deep orange-brown; quinol, dirty greenish-brown; pyrogallol, brown; α-naphthol, dirty green; β-naphthol, dirty brown; cinnamic acid, brown; mandelic acid, yellow.

GOVERNMENT LABORATORIES,
JOHANNESBURG.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Composition of Ripe and Unripe Bananas. R. Reich. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 208-226.)—The author has analysed several different kinds of bananas, and meals prepared from the same, and the following table gives a selection of typical results from the large number of analyses recorded:

	Water.	Water-Soluble Substances.	Invert Sugar.	Sucrose.	Starch.	Nitrogen.	Crude Fibre.	Acidity (as Citric Acid).	Ash.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Dried ripe Gros Michel (Surinam) bananas 1	19·00	73·50	61·86	1·21	3·43	0·64	1·65	1·02	3·30
Dried ripe Gros Michel (Surinam) bananas 2	19·50	72·90	60·28	2·62	3·70	0·64	—	—	3·32
Dried ripe Gros Michel (Surinam) bananas 3	23·20	68·90	60·28	1·29	3·60	0·53	—	—	3·02
Do. do. unripe ...	15·34	9·00	2·24	—	72·45	0·68	2·40	—	2·83
Do. do. unripe, meal	15·40	9·04	2·06	—	71·74	0·69	2·21	—	3·12
Dried ripe "Appel Bacoven" (apple bananas) 1	21·31	64·94	57·68	1·37	7·40	0·46	—	1·28	2·40
Dried ripe "Appel Bacoven" (apple bananas) 2	21·80	66·20	55·36	4·00	5·77	0·45	1·85	1·59	2·44
Do. do. unripe, meal	14·44	5·54	0·55	—	74·15	0·50	3·70	—	2·15
Dried ripe "Pikien Missi-finger Bacoven" 1	23·71	66·20	55·60	3·15	4·80	0·62	1·26	1·16	2·16
Dried ripe "Pikien Missi-finger Bacoven" 2	23·50	63·30	51·56	3·08	9·03	0·62	—	—	2·17
Cooking bananas (plantains)—unripe	75·00	—	—	—	21·32	—	0·22	—	0·48
Do. do. ripe ...	75·00	—	—	—	18·06	—	0·19	—	0·64
Do. do. meal ...	16·20	—	—	—	69·40	—	0·90	—	2·30
Canary bananas — ripe ...	74·75	22·79	9·79	9·96	0·93	—	0·64	0·41	1·12
Do. do. overripe ...	79·16	18·42	11·66	3·90	0·90	—	—	—	0·95
Jamaica bananas — halfripe ...	73·69	19·89	6·72	10·35	4·20	0·18	—	—	0·95
Dried banana skins, unripe ...	6·60	17·79	3·87	—	40·28	0·91	10·10	0·96	11·43
Do. do. ripe ...	7·40	40·42	20·03	—	8·17	1·04	12·66	2·07	13·45

It will be seen from the above results that the starch present in bananas is converted into sucrose and invert sugar during the ripening process (*cf.* ANALYST, 1911, 274).

For the manufacture of banana meals, care is taken that the fruit is collected at a period when the formation of starch is fully developed, but before the saccharification of the starch commences. W. P. S.

Composition of Roquefort Cheese. A. W. Dox. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 239-242.)—The following results were obtained on the analysis of eight samples of Roquefort cheese purchased in New York:

	Water.	Fat.	Protein. N × 6·37.	Ash.	Sodium Chloride.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Maximum	40·10	33·53	23·25	6·81	4·50
Minimum	37·49	31·50	19·94	5·48	3·64
Average	38·61	32·24	21·62	6·19	4·18

The high percentage of sodium chloride distinguishes this class of cheese from Gorgonzola (1·57 per cent.) and Stilton (0·59 per cent.). W. P. S.

Nature of the Poisonous "Cardamom" (Maratti) Fat employed in the Manufacture of "Backa-Margarine." H. Thoms and F. Müller. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 226-236.)—The recent case of poisoning by "backa-margarine" having been found to be due to the presence of maratti fat in the margarine, the author investigated the characters and sources of this fat. It seems that the fat was obtained from an Indian species of *Hydnocarpus*, probably from the most widely distributed member of the species—namely, *Hydnocarpus Kurzii Warburg* (synonym: *Taraktogenos Kurzii King*). The fat has the following constants: m.p., 22° to 24° C.; $[\alpha]_D = +54·0°$ to 58·0°; saponification value, 201 to 212; and an iodine value from 88 to 94·8. It appears to be identical with chaulmugra fat; besides palmitic acid, the fat contains hydnocarpic acid and chaulmugric acid, $C_{18}H_{32}O_2$; m.p. 68·5°. Both maratti fat and chaulmugric acid are poisonous, a dose of 0·04 grm. of the former killing a white mouse within an hour and a half. (*Cf.* ANALYST, 1911, 65.) W. P. S.

Estimation of the Reichert-Meissl Value of Fats by Means of Glycerol-Potassium Hydroxide. H. Kreis. (*Chem. Zeit.*, 1911, **35**, 1053-1054.)—The author states that butter, cocoanut oil, lard, etc., may be saponified for the estimation of the Reichert-Meissl value, by the use of 2 c.c. of 50 per cent. potassium hydroxide solution and 4 c.c. of glycerol. The saponification proceeds more rapidly than when the usual 20 c.c. of glycerol are employed, and the results obtained agree closely. The advantage of using the smaller quantity of glycerol arises from the fact that many specimens of glycerol yield considerable amounts of volatile acid substances, the Reichert-Meissl values of fats having to be corrected accordingly. W. P. S.

Estimation of Fat in Feeding Stuffs by Means of Trichlorethylene. R. Neumann. (*Chem. Zeit.*, 1911, **35**, 1025-1026.)—The use of trichlorethylene is ecommended (*cf.* L. Gowing-Scopes, ANALYST, 1910, **35**, 238) for the estimation of fat in such feeding stuffs as meals, feeding-cakes, etc. Five grms. of the material are shaken for one hour in a stoppered flask with 100 c.c. of the solvent; after being allowed to settle for thirty minutes, the liquid is filtered, 50 c.c. of the filtrate are evaporated in a weighed flask, the residue of fat is dried for one hour at 100° C. and weighed. The results obtained agree well with those yielded by the ordinary extraction process with ether. W. P. S.

Method of Estimating the Quality of Wheaten Flour. L. von Liebermann and V. Andriska. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 291-293.)—The quality of wheaten flour as regards the quantity of bran substance present may be ascertained by shaking the flour with chloroform, and observing the colour of the portion which floats on the surface of the chloroform. The test may be rendered quantitative in the following manner: One grm. of the flour is shaken in a tube with 10 c.c. of chloroform, and the mixture is allowed to stand for one hour. A layer then forms on the surface of the chloroform, and its depth of colour is compared with the colours of the layers produced when mixtures of finest white flour with variable proportions of bran are subjected to similar treatment; these mixtures may conveniently contain quantities of washed bran ranging from 0 to 2 per cent. The colour of the layers should be observed from above. W. P. S.

Composition of the So-called Gallisin [Glucosin] occurring in Commercial Starch Syrup. J. Gatterbauer. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 265-290.)—That portion of commercial starch syrup which resists fermentation, and to which the name "gallisin" has been applied, consists of a small quantity of maltose together with a new carbohydrate which has the properties of a disaccharide; dextrin is not present. This carbohydrate, or "glucosin," yields only dextrose on hydrolysis, and may be regarded as an isomer of maltose. It is obtained from completely fermented starch syrup as a viscous liquid which, by treatment with absolute alcohol and ether, may be converted into a white or yellowish-white powder. "Glucosin" has $[\alpha]_D = +100°$, is fermented slowly by beer-yeast, and is converted by yeast-maltase and by emulsin into dextrose. Mineral acids and oxalic acid also convert "glucosin" into dextrose, but with high concentrations of mineral acids the reaction is reversible. In fact, the new carbohydrate is formed by the action of mineral acids on dextrose during the manufacture of starch syrup. The phenylosazone of "glucosin" is a red-brown crystalline powder melting at 97° to 100° C.; the *p*-nitrophenylosazone melts at 240° C., and yields a blue coloration when treated with sodium hydroxide. "Glucosin" also yields amorphous esters with benzoyl chloride, *p*-chlorobenzoyl chloride, *m*-nitrobenzoyl chloride, and benzene sulphochloride. W. P. S.

Estimation of Sulphurous Acid in Glucose Syrup. O. Claassen. (*Chem. Zeit.*, 1911, **35**, 1065-1066.)—The usual addition of calcium carbonate in estimating sulphurous acid by distillation with phosphoric acid and absorption in potassium iodide solution is shown to be an unnecessary complication. Thus, in comparative estimations with and without the addition of 3 grms. of calcium carbonate, the amounts of sulphur dioxide found in a glucose syrup were 0·0056 and 0·0050 per cent. respectively. No differences in the results were produced by the substitution of acetic acid for phosphoric acid. C. A. M.

Some Reactions of Gum Kino. J. L. Simonsen. (*J. Chem. Soc.*, 1911, **99**, 1530-1535.)—The author was unable to isolate the principle kinoin by the action of hydrochloric acid on kino. Catechol was, however, detected, and this is generally present in small quantity in kino. Kino is not glucosidic in character. It was found to be readily methylated by treatment with methyl sulphate and potassium hydroxide. (No methoxy-group was discovered in kino.) The methyl ether, $C_{15}H_{11}O_4(OMe)_3$, was obtained as a non-crystalline, nearly colourless powder, and the acetate, a brown powder, was of similar characters and corresponding formula. By oxidation in the cold with potassium permanganate, the methyl ether yielded veratric acid as the only product; by fusion with potassium hydroxide, protocatechuic acid and catechol were obtained, but no phloroglucinol. On oxidation with 10 per cent. nitric acid, oxalic acid and ammonium quadroxalate were formed, the latter probably by means of the strong reducing action of kino on the acid with production of ammonia.

A. R. T.

Estimation of Nicotine in Nicotine Solutions and Tobacco Extracts. R. M. Chapin. (*U.S. Dept. of Agriculture, Bureau of Animal Industry*, Bull. No. 133, 1911; through *Chem. Zentralbl.*, 1911, II., 799-800.)—The results of a critical examination of various methods used for the estimation of nicotine are recorded. Kissling's method was found to yield accurate results; as, however, all alkaline substances present in the distillate are counted as nicotine, it is probable that the correct results obtained are due to a balancing of errors. According to the directions of the American Association of Official Agricultural Chemists, the method is carried out as follows: Five grms. of tobacco extract, or 20 grms. of finely divided tobacco dried at 60° C., are mixed with 10 c.c. of alcoholic sodium hydroxide solution (6 grms. of sodium hydroxide, 40 c.c. of water, and 60 c.c. of 90 per cent. alcohol); in the case of tobacco extracts, sufficient calcium carbonate is also added, so as to convert the mixture into a moist mass. This is extracted with ether for five hours in a Soxhlet apparatus, the ethereal solution is evaporated at a low temperature, the residue is dissolved in 50 c.c. of 4 per cent. sodium hydroxide solution, transferred to a flask, and steam-distilled until 500 c.c. of distillate have been collected; at the end of the distillation the flask should contain only about 15 c.c. of liquid. The distillate is then titrated with $\frac{N}{1}$ sulphuric acid, using phenacetolin as indicator.

Toth's method (ANALYST, 1902, **27**, 12) also yields trustworthy results, although the danger that non-volatile alkaloids, if present, would be included in the nicotine result.

The silicotungstic acid process, proposed by Bertrand and Javillier (ANALYST, 1909, **34**, 219), is considered by the author to be the most convenient and accurate method for the estimation of nicotine. The procedure recommended is as follows: A quantity of the sample, corresponding with from 1 to 2 grms. of nicotine, is mixed in a flask with 1·5 grms. of paraffin, a small quantity of pumice-stone, and 10 c.c. of sodium hydroxide solution (1 : 2), and submitted to steam-distillation until a few c.c. of the distillate no longer yield a turbidity with silicotungstic acid. The distillate is received in a vessel containing 10 c.c. of hydrochloric acid (1 : 4), and at the end of the distillation is diluted to a definite volume and filtered. A quantity of the filtrate corresponding with about 0·1 grm. of nicotine is then treated with a further quantity of hydrochloric acid, 3 c.c. of the latter (1 : 4) being added for each 100 c.c. of the solution, and 10 c.c., or an excess, of 12 per cent. silicotungstic acid are added. After the lapse of eleven hours the precipitate is collected on a filter, washed with water containing 1 c.c. of hydrochloric acid per litre, and ignited in a platinum crucible. The weight of the residue multiplied by 0·114 gives the quantity of nicotine in the precipitate. The precipitate may also be collected in a Gooch crucible, dried at 125° C., and weighed as anhydrous nicotine silicotungstate ($2C_{10}H_{14}N_2.2H_2O.SiO_2.12WO_3$). W. P. S.

Analysis of Tobacco Juice and Extracts, and of Nicotine Salts, by the Method of Ulex. Essner. (*Ann. Chim. anal. appl.*, 1911, **16**, 339-341.)—The method of Ulex, the accuracy of which has been called in question by Koenig (ANALYST, 1911, 106, 348), gives closely concordant results when applied in the following manner: About 10 grms. of the juice or extract are mixed with soda-lime prepared by slaking quicklime with sodium hydroxide solution (sp. gr. 1·33), leaving a slight excess of lime, mixing one part of the mass with four parts of plaster, grinding the whole in a mortar, and sifting the powder through a sieve with 220 meshes to the sq. cm. The mixture of tobacco juice and soda-lime should also be sifted, any residue left being ground up again until the whole is reduced to a fine powder. This is exposed for about an hour in a wide vessel placed in a desiccator over sulphuric acid to facilitate the evaporation of ammonia and ammoniacal compounds prior to the distillation, after which it is transferred to a 3-litre flask containing 2½ litres of boiled water. The evaporating basin is rinsed with a few drops of hydrochloric acid, which are also added to the main liquid. About 10 grms. of sodium hydroxide and 10 to 15 grms. of paraffin wax (to prevent bumping) are now added, and the liquid distilled so as to obtain six fractions of 1,000, 500, 200, 100, 100, and 100 c.c. of distillate. Each of these fractions is separately titrated with $\frac{N}{2}$ hydrochloric acid (1 c.c. = 0·081 grm. of nicotine), with turmeric as indicator, and each result added to the preceding one. From 60 to 70 per cent. of the nicotine passes over with the first fraction, and from 85 to 90 per cent. in the first 1,500 c.c. In this way the author has estimated nicotine within 1 per cent. in concentrated tobacco extracts containing 100 to 125 grms. of the alkaloid per litre, and varying in sp. gr. from 1·060 to 1·450; whereas by Schloesing's method deviations of 10 to 15 grms. were obtained in duplicate estimations, even when the proportion of alkali was increased beyond the specified amount.

C. A. M.

Estimation of Tartaric Acid in Apples, Pears, Cider, and Perry. G. Warcollier. (*Ann. Falsific.*, 1911, 4, 485-490.)—Kling's method of estimating tartaric acid by precipitation as calcium racemate gives very accurate results. This racemate, when dissolved in hot dilute sulphuric acid, gives a quantitative reduction of potassium permanganate solution. Examined by this method, neither cider nor perry, nor the ripe or unripe fruits from which they are prepared, contained any trace of tartaric acid. C. A. M.

Detection of the Substitution of Spirit for Aged Whisky. A. B. Adams. (*J. Ind. Eng. Chem.*, 1911, **3**, 647-655.)—An account is given of the chemical and other methods employed for ascertaining whether barrels of whisky, drawn from bond and bearing Government stamps and seals indicating the quality of the whisky, had been partially emptied of their contents and refilled with other spirit by the dealers. In dealing with the cases mentioned (these were the subject of a law case, in which the findings of the analyst were upheld), samples were obtained from the distilleries, etc., as nearly as possible representing the age and quality of the suspected samples; other samples of new and old whisky were also examined for the purpose of comparison, and with the object of ascertaining the rate of change in the character and quantity of the different constituents of the whiskies during the ageing process. It was found that there is a general tendency for the alcoholic strength, colour, solids, and secondary substances, to increase with storage; this increase is uniform and comparable in barrels of proximately the same age. The barrel seems to have no other influence than to cause a slight variation in the depth of colour and amount of aldehydes. The quantity of furfural depends almost entirely on the kind of barrel and the time of storage. In all cases the composition of the colour extracted from the barrels is the same, never less than 70 per cent. insoluble in water, or the same percentage soluble in amyl alcohol. The suspected samples were not similar in composition to any of the genuine comparison samples, except as regards alcoholic strength and depth of colour. The proportions of esters, acids, and colour, yielded the most useful data in judging the quality of the samples, the content of total solids, higher alcohols, aldehydes, and furfural, affording only confirmatory evidence. W. P. S.

Analysis of Turpentine Oil. C. Blarez. (*Ann. Chim. anal. appl.*, 1911, **16**, 328-336.)—One hundred c.c. of the oil are distilled so that 80 c.c. pass over during the first twelve to fifteen minutes, and four successive fractions of 20 c.c. each of the distillate are collected, whilst the residue in the flask constitutes a fifth fraction. French turpentine oils (from *Pinus maritima*) showed the following characteristics: Sp. gr. at 15° C., 0·8660 to 0·8720; polarimeter reading, −140° to −143°; turbidity temperature with aniline, (2 c.c. + 2 c.c.), 170 to 180 (sometimes 160 in the case of old oils); refractive index at 15° C., 1·4720 to 1·4730; and acid value, 1·5. Greek, Algerian, and Provence oils (from *P. alepensis*) had a lower sp. gr., though always superior to 0·8542, and gave a polarimetric reading ranging from +177° to +190°; whilst American oils (from *P. palustris*, etc.) had sp. grs. intermediate between those of the other oils, and showed sometimes a dextro- and sometimes a lævo-rotation. The effect of the addition of 5 per cent. of various common adulterants upon a pure oil of turpentine is shown in the following table of results:

Adulterants added to Pure Turpentine Oil in the Proportion of 5 per Cent.	Pure Oil.	Leaf-bud Turpentine (*Gemme terebenthine*).	Rosin.	Crude Essential Oil (*Essence vive brute*).	Rosin Oil.	Bleached Rosin Oil.	Rectified Benzene.	Mineral Essence	White Spirit.	Petroleum Lamp Oil.
Sp. Gr. at 15° C.										
Original product	0·8683	0·8717	0·8794	0·8714	0·8709	0·8739	0·8716	0·8628	0·8648	0·8689
First fraction	0·8634	0·8649	0·8642	0·8635	0·8636	0·8637	0·8687	0·8472	0·8496	0·8669
Fifth „	0·8634	0·8961	0·9220	0·9018	0·8909	0·9028	0·8860	0·8909	0·8896	0·8813
Polarimetric Reading.										
Original product	140·8	142·0	125·5	126·8	132·0	112·5	134·5	133·0	133·0	132·5
First fraction	152·5	153·5	152·0	151·2	151·7	152·0	123·0	126·0	135·5	148·5
Second „	150·7	151·8	149·8	150·0	150·0	150·0	146·2	149·0	142·0	148·5
Third „	148·0	149·0	147·0	146·3	147·0	147·5	147·3	149·0	143·7	145·0
Fourth „	143·3	144·0	141·0	139·7	141·3	141·0	143·0	145·0	139·0	140·0
Fifth „	107·0	106·7	29·0	58·0	69·0	12·5	104·0	104·0	96·0	83·0
Turbidity Temperature (Tenths of a Degree) with Aniline.										
Original product	173	125	116	165	171	170	125	189	189	198
First fraction	210	204	205	208	206	205	15	257	237	218
Second „	205	200	200	205	200	200	180	216	218	212
Third „	195	195	192	200	190	190	195	200	202	202
Fourth „	180	181	175	169	176	175	182	185	184	190
Fifth „	73	155	192	40	87	88	75	88	106	170
$[N]_{D\ 15^\circ\ C.}$										
Original product	1·4729	1·4759	1·4767	1·4752	1·4750	1·4763	1·4743	1·4702	1·4700	1·4743
First fraction	1·4704	1·4707	1·4708	1·4707	1·4702	1·4712	1·4748	1·4592	1·4649	1·4688
Second „	1·4706	1·4709	1·4710	1·4709	1·4704	1·4713	1·4718	1·4690	1·4683	1·4693
Third „	1·4711	1·4712	1·4713	1·4713	1·4706	1·4719	1·4718	1·4700	1·4694	1·4698
Fourth „	1·4716	1·4716	1·4722	1·4721	1·4716	1·4725	1·4720	1·4708	1·4703	1·4708
Fifth „	1·4800	1·4937	1·4994	1·4914	1·4907	1·4905	1·4807	1·4793	1·4788	1·4789
Acid Value	1·00	7·84	9·52	1·56	1·00	1·00	0·95	0·95	0·95	0·95

C. A. M.

Composition of Sauerkraut. E. Feder. (*Zeitsch. Untersuch. Nahr. Genussm.* 1911, **22**, 295-296.)—Analyses of this substance yielded the following results :

	Water.	Proteins.	Fat.	Lactic Acid.	Dextrose.	Mannose.	Crude Fibre.	Ash.	Sodium Chloride.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
1	90·84	1·68	0·28	1·75	—	0·80	0·92	1·40	0·78
2	88·00	1·31	0·38	1·22	0·80	0·97	0·87	4·04	3·31
3	89·00	1·46	0·38	1·78	—	1·16	1·02	2·20	1·45
4	88·40	1·37	0·31	1·33	1·31	1·01	0·98	3·05	2·34

When an aqueous extract of sauerkraut is poured on the surface of concentrated sulphuric acid, a violet-coloured ring is formed at the junction of the two liquids; the coloration is intensified if the extract be mixed previously with a small quantity of milk, the reaction in this case being similar to that yielded by formaldehyde. Concentrated hydrochloric acid also yields a violet coloration. Mannose appears to be an important constituent of sauerkraut, its quantity amounting to about 10 per cent. of the dry solids. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Method for the Estimation of Acetone in Animal Fluids. H. Scott-Wilson. (*J. of Physiol.*, 1911, **42**, 444-470; through *Chem. Zentralbl.*, 1911, II., 903-904.)—For the estimation of acetone in urine and other animal liquids, the following modification of Marsh and Struthers' method (*J. Chem. Soc.*, 1906, **87**, 1878) has been worked out: 100 c.c. of urine (or less if the quantity of acetone be abnormally high) are placed in a boiling-flask with 25 grms. of anhydrous sodium sulphate and 1 c.c. of concentrated sulphuric acid. The flask is provided with a leading tube communicating with the outside, which is closed during the distillation, also with another tube leading into a second flask. This flask contains 10 c.c. of a 40 per cent. sodium hydroxide solution, and is connected through a reflux condenser with a flask containing the reagent. The reagent consists of a solution of 0·5 grm. of mercuric cyanide, 9 grms. of sodium hydroxide in 60 c.c. of water and 20 c.c. of a 0·7268 per cent. solution of silver nitrate; if more than 0·4 mgrm. of acetone be expected, the quantity of the reagent should be increased. On boiling the urine, the free acetone, together with that derived from any aceto-acetic acid, is driven over, through the previously heated soda solution in the second flask, which holds back phenols and volatile acids, into the reagent flask. Distillation is continued for five minutes after the first appearance of a turbidity in the mercury solution; prolonged distillation leads to a further production of acetone, owing to secondary changes. Ten minutes after the distillation has been stopped, a few drops of an emulsion of talc are added, and the liquid is filtered through a Gooch crucible. The precipitate together with the filter-paper is placed in a flask with about 10 c.c. of a mixture of 40 per cent. of nitric acid, 5 per cent. of sulphuric acid, and 55 per cent. of water;

1 c.c. of $\frac{N}{5}$ permanganate solution is added, and the liquid is boiled. The colour should disappear in one or two minutes; if it disappears in a few seconds, a further 1 c.c. of permanganate is necessary. The oxidised liquid is treated with 2 c.c. of saturated iron-alum solution and titrated with standard thiocyanate (1 c.c. = 1 mgrm. of mercury), until a brown colour is produced: 1 mgrm. of mercury corresponds to 0·058 mgrm. of acetone. If the urine has suffered decomposition by standing for twenty-four hours, the quantity of acetone may be increased by 7 to 21 per cent. in consequence of secondary reactions. The mercury reagent may also be used for the qualitative detection of an abnormal proportion of acetone in the atmosphere. The daily quantity of acetone excreted by the normal organism ranges from 2 to 4 mgrms.

J. F. B.

Absorption of Arsenic by Sugar Beets. H. Remmier. (*Chem. Zeit.*, 1911, **35**, 977-979.)—For the destruction of the horse-beetle on growing beet, the application of a spray composed of Schweinfurt green and lime to the young plants, at the end of May, has been recommended. The author has conducted experiments to ascertain whether, under these conditions, the presence of arsenic in poisonous quantities can be detected in the leaves or roots at the time of the harvest. The results showed that the beet root is indeed capable of taking up arsenic in this manner, and that the quantity of arsenic absorbed increases with the quantity of Schweinfurt green applied. Exactly by what process this absorption takes place is not yet determined, nor is the exact form of the arsenic compound in the plant known. But assuming it exists entirely in the most poisonous form, the quantity present in the plants which have been treated with the prescribed proportions of Schweinfurt green is so small that no evil results are to be feared. For instance, from 1 kilo of the roots an arsenic mirror corresponding to 0·005 mgrm. of the oxide was obtained; from 500 grms. of the leaves no mirror was produced. Beets which had been treated with an excess of the green, temporarily injurious to vegetation, showed 0·08 mgrm. of arsenious oxide per kilo, and the leaves showed 0·25 mgrm. per kilo. It is remarked that even if traces of arsenic were, by some accidental over-treatment, to appear in the juice, they would certainly be precipitated by the defecation lime; in fact, defecation lime itself may normally contain more arsenic than is likely to be present in the juice of treated beets. J. F. B.

Detection of Blood. O. von Fürth. (*Zeitsch. angew. Chem.*, 1911, **24**, 1625-1628.)—For the identification of blood when present in traces on paper, fabrics, etc., the author recommends the use of a method which is a combination of tests described by Leers (treatment with sodium hydroxide and extraction of the blood-colouring matters with pyridine) and by Adler (leucomalachite green test; *cf.* ANALYST, 1911, 284). The piece of fabric containing the stain to be tested for blood is heated for a minute or so with a few drops of 50 per cent. potassium hydroxide solution and a small quantity of alcohol. After cooling, the solution is transferred to a small separating funnel and shaken with a few c.c. of pyridine; when separation has taken place, the alkaline liquid is drawn off, and the pyridine layer is shaken with a few c.c. of 50 per cent. potassium hydroxide solution. A portion of the pyridine solution may now be examined spectroscopically, whilst another portion of about 1 c.c. is placed on a filter-paper on a glass plate and treated with a few drops of leucomalachite green solution containing 1 per cent. of hydrogen peroxide. A green coloration developes if blood is present. The leucomalachite green reagent is prepared by dissolving 1 grm. of the substance in 50 c.c. of glacial acetic acid, diluting with water to a volume of 500 c.c., and shaking the solution with chloroform to remove any green colour. When carried out in this manner the test is stated to be very sensitive, and to be free from certain objections (interference of other substances) belonging to the original methods of applying the tests. Potassium permanganate and lead peroxide are the only substances which must be removed before the test is applied; for this purpose the fabric should be treated with a small quantity of 50 per cent. hydrazine solution, and then well washed before being heated with the potassium hydroxide solution. Only when blood has been heated to temperatures above 220° C. does it fail to give a positive reaction with the test.

W. P. S.

Hæmochromogen and the Spectroscopic Differentiation between Oxyhæmoglobin and Carbon Monoxide Hæmoglobin. F. Michel. (*Chem. Zeit.*, 1911, **35**, 996.)—The best reagent for reducing the colouring matter of the blood into hæmochromogen is an alcoholic alkaline solution of sodium hydrosulphite prepared by dissolving 4 grms. of potassium hydroxide in 75 c.c. of water, adding 3 grms. of fresh sodium hydrosulphite ($Na_2S_2O_4$), diluting the solution with 40 c.c. of 98 per cent. alcohol, and filtering the liquid rapidly into a stoppered flask. On adding to the solution of blood a few drops to a few c.c. of this reagent, the characteristic spectrum of hæmochromogen is obtained even in the cold. On heating the liquid a broad absorption band appears in the case of concentrated solutions, and the spectrum gradually disappears. It may, however, be restored by the addition of 2 or 3 drops of pure pyridine, the liquid also becoming red again. On now shaking the cold solution, the red colour and hæmochromogen spectrum disappear once more, but may be restored on heating the tube. This behaviour is characteristic of blood. In the case of blood-stains upon linen, paper, etc., it is frequently sufficient to apply a few drops of the reagent and to examine the spot between two glass slips by means of transmitted light. As a confirmatory test or in case of a negative result the stains are heated in a test-tube with about 4 to 6 drops of the reagent and 1 or 2 drops of pyridine, a similar course being adopted for the examination of blood-stains on rusty iron. For the detection of minute quantities of carbon monoxide-hæmoglobin, a few drops of the blood are diluted with water, and a few c.c. of the reagent and a few drops of pyridine added. The colour of the liquid will become an intense yellowish-red, and only the oxyhæmoglobin will have been converted into hæmochromogen, until the liquid is heated, when the carbon monoxide-hæmoglobin spectrum will also give place to that of hæmochromogen.

C. A. M.

Estimation of Indophenol Oxydase of Animal Tissues. H. M. Vernon. (*J. Physiol.*, 1911, **42**, 402-407; through *Chem. Zentralbl.*, 1911, II., 796-797.)—Formation of indophenol takes place when the tissues of a rat or rabbit are mixed with α-naphthol, *p*-phenylenediamine, and sodium carbonate; with *M*/100 α-naphthol and phenylenediamine solution, the quantity of indophenol formed is proportional to the square of the amount of enzyme, with *M*/150 solution it is directly proportional to the quantity of enzyme, and with *M*/400 solution is proportional to the square root of the quantity of enzyme. The estimation may be carried out by mixing quantities of 1 grm. of the tissue in Petri dishes with 5 c.c. of *M*/100, *M*/150, and *M*/400 α-naphthol and *p*-phenylenediamine solutions containing 0·25 per cent. of sodium carbonate; at the end of a few hours the mixtures are digested for twenty-five minutes with 97 per cent. alcohol, a portion of the liquid is filtered, and the coloration is compared with that of standard solutions. The latter are prepared by mixing 1 part of the above reagent solutions with 200 parts of 50 per cent. alcohol, adding 1 part of 0·5 per cent. calcium hypochlorite solution, and allowing the mixtures to stand for twenty-four hours. The tissues of the rat are richer in oxydase than are those of the rabbit. The heart muscle contains most oxydase, then follow the tongue muscles, the diaphragm, the renal cortex, and the grey substance of the brain.

W. P. S.

Detection of Active Pepsin in the Intestines by Means of Elastin. E. Abderhalden and C. Meyer. (*Zeitsch. physiol. Chem.*, 1911, **74**, 67-100.)—Considerable quantities of active pepsin were found to be present in the intestines of animals, and this ferment seems to play an important part, in conjunction with trypsin and erepsin, in the digestion of proteins after the latter have passed the stomach. The method employed in detecting the presence of pepsin depended on the property, possessed by proteins, of being capable of absorbing pepsin; elastin was used in the experiments, quantities of it being saturated with $\frac{N}{10}$ hydrochloric acid and then immersed in the intestinal contents. After the lapse of two hours the elastin was removed, washed, and kept, together with a small quantity of water, for forty-eight hours at a temperature of 37° C. The optical activity of the solution and its behaviour towards the biuret reaction were then determined. W. P. S.

Rapid Gravimetric Method for the Estimation of Albumin in Urine. E. Simonot. (*Bull. Soc. Chim.*, 1911, [IV.], **9**, 839-842.)—Most of the methods for the gravimetric estimation of albumin are not quite satisfactory, either because of incomplete coagulation, slow filtration, or a variable coefficient; in some cases, also, the coagulum slowly disintegrates during washing. The author finds that metaphosphoric acid coagulates the albumin with results satisfactory in all respects. It is preferably liberated *in situ*, by adding a solution of sodium metaphosphate to the urine and treating with an acid. A 5 per cent. solution of sodium metaphosphate is prepared by dissolving 5·7 grms. of the finely powdered salt in 50 c.c. of hot water, boiling for five minutes, adding 50 c.c. of cold water, cooling rapidly to 15° C., and making up to 100 c.c.; the quantity of metaphosphate converted into phosphate by hydrolysis under these conditions is negligible. The urine is first tested approximately with this reagent by the diaphanometric method of Denigès (*Chimie analytique*, 3rd edition, 1907, 1011), and it should contain over 0·5 grm. of albumin per litre. A quantity of urine containing between 0·08 and 0·15 grm. of albumin is measured out, and its volume is made up to at least 100 c.c. It is placed in a capsule and heated on the boiling-water bath for ten minutes if 100 c.c. are taken, or for fifteen minutes if the volume is 200 c.c. Then are added without stirring 5 c.c. of a 5 per cent. solution of sodium metaphosphate and 1 c.c. of pure hydrochloric acid for every 100 c.c. of urine, and heating is continued for a further five or ten minutes. The coagulated albumin is deposited for the most part on the bottom of the dish. It is collected on a tared filter, dried at 110° to 120° C., care being taken to decant the liquid off first, washed with boiling water until the filtrate no longer reacts with silver nitrate, then with alcohol, and lastly with ether. The precipitate is drained by pressing between filter-paper, then dried at 110° to 120° C. and weighed. The weight multiplied by the factor 0·88, gives the weight of albumin in the urine. J. F. B.

Detection of Mercury in Urine. E. Salkowski. (*Zeitsch. physiol. Chem.*, 1911, **73**, 401-406.)—In the method described the mercury is deposited on copper, volatilised from the latter on to the sides of a glass tube, and then converted into mercuric iodide by treatment with iodine vapour. The urine is first evaporated and oxidised by means of hydrochloric acid and potassium chlorate, and filtered if necessary, in order to obtain a final solution which is about 10 c.c. in volume. Two or three strips of copper-foil, about 50 mm. long and 10 mm. wide, are immersed in this solution for an hour and a half, then removed, washed with water, alcohol, and ether. The dry strips are next heated in a dry test-tube until the mercury has sublimed on to the sides of the tube; the latter is then cooled and the strips are removed. A minute fragment of iodine is now dropped into the tube, which is placed in a boiling-water-bath until the excess of iodine has been expelled; a current of air may be blown into the tube to aid in the removal of the vapours. Should mercury be present in the urine under examination, a red mirror of mercuric iodide will be formed inside the test-tube. The test will detect as little as 0·19 mgrm. of mercury in 500 c.c. of urine. W. P. S.

Separation and Identification of Urobilin. C. Carrez. (*Ann. Chim. anal. appl.*, 1911, **16**, 337-339.)—Forty-five c.c. of the urine under examination are shaken with 30 c.c. of Denigès' mercuric sulphate reagent (ANALYST, 1898, **23**, 216) and filtered, and 50 c.c. of the filtrate (=30 c.c. of urine) shaken with about 1 grm. of talc or silica, to precipitate the urobilin. The precipitate is washed with water and tested for urobilin. Thus it may be directly treated with 5 to 6 c.c. of Oliviero's reagent (zinc chloride 10 grms. dissolved in about 30 grms. of ammonia solution, and mixed with 80 grms. of 90 per cent. alcohol, and 20 grms. of ethyl acetate), which gives an immediate green fluorescence with urobilin, the liquid showing a characteristic spectrum with an absorption-band at γ. Or the precipitate may be treated with 10 c.c. of alcohol acidified with 5 per cent. of hydrochloric acid, and the extract shaken with 5 to 6 c.c. of chloroform and 100 to 150 c.c. of water. The chloroform extract is separated and tested with a 0·1 per cent. alcoholic solution of zinc acetate, which is added drop by drop until a clear solution is obtained. In the presence of urobilin the characteristic fluorescence and spectrum will then be observed. (*Cf.* ANALYST, 1911, 223.) C. A. M.

ORGANIC ANALYSIS.

New Method of Elementary Analysis of Organic Substances containing Carbon, Hydrogen, Oxygen, and Nitrogen. J. A. A. Auzies. (*Bull. Soc. Chim.*, 1911, [IV.], 9, 814-815.)—The combustion-tube is charged with plugs of asbestos or cotton impregnated with thoria, in place of the usual copper oxide; otherwise it is fitted up in the ordinary way. The absorption vessels contain calcium chloride to absorb the water, a titrated solution of cuprous chloride in hydrochloric acid to absorb nitrogen peroxide, and potash for the carbon dioxide. The vapours of nitrogen peroxide when absorbed by the cuprous chloride produce a coloration which is proportional to the quantity absorbed. This coloration is then destroyed by the addition of a titrated solution of stannous chloride, according to reactions in which $SnCl_2$ is equivalent to $4NO_2$. The thoria cotton is prepared by steeping the cotton in a solution of a thorium salt, then in a solution of ammonia, which precipitates the hydroxide, and finally incinerating. J. F. B.

Analysis of a Substance containing Carbon, Hydrogen, Oxygen, Nitrogen, and Sulphur. J. A. A. Auzies. (*Bull. Soc. Chim.*, 1911, [IV.], 9, 815.)—In the case of a substance containing sulphur, the gases, after passing over the thoria (see preceding abstract), are conducted over lead peroxide. It is necessary to separate the lead peroxide from the thoria—for instance, by interposing a plug of asbestos, since means must be provided for removing the lead peroxide quantitatively, prior to dissolving in hydrochloric acid for the estimation of the sulphate. The best method is to pack the lead peroxide in a special cylindrical boat which slides easily into the tube, and can be withdrawn after the analysis, the lead sulphate being estimated in the ordinary way. J. F. B.

Analysis of a Substance containing Carbon, Hydrogen, Oxygen, Nitrogen, Sulphur, Chlorine, Bromine, and Iodine. J. A. A. Auzies. (*Bull. Soc. Chim.*, 1911, [IV.], 9, 815-819.)—In the case of a substance containing all the above elements, the tube is fitted up with absorption vessels containing calcium chloride, cuprous chloride, and potassium hydroxide. These account for the carbon, hydrogen, and nitrogen. The sulphur is absorbed by lead peroxide contained in the combustion-tube. In a similar manner the halogens are absorbed by means of silver chromate, also contained in a special cylindrical boat. After the combustion, the silver chromate is treated with ammonia. The ammoniacal solution contains the silver chloride and the excess of silver chromate. The filtered liquid is neutralised by acetic acid, which precipitates the silver salts, and the chloride is then re-dissolved in potassium cyanide and estimated by known methods. The residue from the ammoniacal treatment contains silver bromide and silver iodide. This is washed on the filter and treated with potassium cyanide solution, which dissolves the iodide, leaving the bromide on the filter. The complete simultaneous estimation of all these elements in a mixture of substances has been carried out with satisfactory results. The following directions are given for preparing and standardising the cuprous chloride solution used for absorbing the nitrogen peroxide: 35 to 40 grms. of copper sulphate are dissolved in a litre of water; a stannous chloride solution is also prepared by dissolving 20 grms. of the salt in 300 c.c. of water and diluting to 1 litre. Ten c.c. of the copper solution are then boiled with 25 c.c. of hydrochloric acid, and the stannous chloride is run in until decolorisation is complete. Three c.c. of a solution of pure potassium nitrate containing 0·01 grm. of N_2O_5 are then added, and the coloured liquid is again exactly decolorised by the stannous chloride, which is thereby standardised in terms of nitric acid. J. F. B.

Analysis of Beeswax, with Special Reference to East Indian and the So-called China Beeswax. G. Buchner. (*Zeitsch. öffentl. Chem.*, 1911, **17**, 225-227; through *Chem. Zentralbl.*, 1911, II, 729.)—When pure beeswax is saponified, the alcohol evaporated, and the residue treated with 250 c.c. of boiling water, a clear solution is usually obtained; certain waxes (East Indian, China, etc.) yield turbid solutions with the separation of amorphous substances, and the following tests are described for distinguishing these waxes from those adulterated with paraffin, which also yield turbid solutions. The amorphous matter is separated and boiled with acetic anhydride; a clear solution which does not yield a precipitate on cooling, indicates the presence of aliphatic alcohols, and cetyl alcohol. A crystalline precipitate on cooling denotes the presence of cholesterol, phytosterol, or myricyl alcohol, whilst a turbid solution in hot acetic anhydride indicates paraffin. The presence of a trace of alcohol retards the separation of wax alcohols from the aqueous mixture, but aids in the separation of paraffin. Estimations of glycerol in pure beeswaxes gave results which corresponded with the presence of from 0·08 to 0·1 per cent. of glycerides in the waxes. W. P. S.

Colorimetric Method for the Quantitative Estimation of Enols in Allelotropic Mixtures. L. Knorr and H. Schubert. (*Ber.*, 1911, **44**, 2772-2778). —The reaction of enolic compounds with ferric chloride plays an important part in the study of cases of desmotropy for the detection of the point at which the ketonic form begins to change into the enolic form. By the use of suitable standards and a colorimeter this reaction may be employed in a quantitative sense for the study of the equilibria at different temperatures and in different solvents. The maximum colour intensity is attained when 1 molecule of ferric chloride ($FeCl_3$) reacts with 1 molecule of the enol. A convenient standard is obtained by mixing a freshly prepared solution of the pure enolic form with an equimolecular quantity of sublimed dry ferric chloride. This solution is compared with an equally concentrated solution of the enol-ketone mixture to be examined containing the same quantity of ferric chloride as the standard solution. The above standard answers well in all cases where the enolic form can be obtained in the pure state, and is sufficiently stable in solution during the time of the experiment. In other cases standards may be made from the readily isolated ferric-enol salts of general formula FeR_3, which give maximum colorations when mixed with 2 molecules of ferric chloride and 3 molecules of hydrochloric acid. The method of preparing these salts is described in the paper. In certain other cases it is possible to use as standards stable solutions in which the equilibrium has been reached and the percentage of enol determined by one of the other standards.

The coloured solutions in organic solvents are generally very stable. The method has been used for the study of certain cases of desmotropy, that of methyl mesityloxidoxalate being described at length in the paper. About 0·2 grm. of the sample is weighed out, treated with the equivalent quantity of ferric chloride in alcoholic solution, made up to 50 c.c. with alcohol, and examined colorimetrically. J. F. B.

Specifications and Sampling of Fuel Oil and Natural Gas. I. C. Allen. (*U.S. Bureau of Mines; Technical Paper* No. 3, 1911, 1-13.)—*Specification.*—Fuel oil should be either a natural homogeneous oil or a homogeneous residue from a natural oil. If the latter, all low-flashing constituents should have been distilled off. The oil should not consist of a light oil and a heavy residue mixed in such proportions as to give the density required. The oil should not have been "burned" by distillation at too high a temperature, nor heated so as to cause separation of flecks of carbonaceous matter. The flash-point must not be below 60° C., in a closed Abel-Pensky or Pensky-Marten's tester. The limits of sp. gr. are from 0·85 to 0·96 at 15° C. The oil should be free from solid or semi-solid bodies, be mobile, and flow readily at ordinary temperatures, under a head of 1 foot of oil, through a 4-inch pipe 10 feet in length; it should not congeal nor become too sluggish to flow at 0° C. The oil should have a calorific value of not less than 10,000 calories per grm. (18,000 B. T. units per pound), 10,250 calories to be the standard. A bonus is paid, or a penalty inflicted according as the fuel is delivered above or below the standard. The oil should be rejected if it contain more than 2 per cent. of water or 1 per cent. of sulphur, and it should not contain more than a trace of sand, clay, or dirt. The oil should be described by its commercial name, and a statement made as to whether it is a crude oil, refinery residue, or a distillate.

Calculation of Weight of Oil.—The coefficient of expansion of ordinary fuel oil of asphaltic base is about 0·0006 per 1° C. This temperature correction having been made, the weight of 1 gallon in pounds is 8·3316 times the corrected sp. gr., and the weight of 1 cubic foot in pounds is 62·3425 times the corrected sp. gr. at 15° C.

Sampling.—In sampling with a dipper from a waggon, a pint of oil is taken at regular intervals from the flowing oil, twelve or more lots being taken and thoroughly mixed together, the final sample being stored in a tightly soldered can. An iron container absorbs sulphur, and must not be used. A continuous sample may be taken from a cock on the underside of the delivery-pipe during the entire time of discharge.

In sampling from a large tank, the detection of notable quantities of sediment should cause the rejection of the oil. If the oil has stood for a time sufficient to form layers of varying density, it should be sampled by means of a tinned tube of 1 inch diameter, the tube carrying a conical wooden or cork plug, with an attached wire passing up through the tube. The tube, open at both ends, is carefully lowered into the oil, and when the bottom is reached a tug on the wire will cause the plug to close the tube, and this can be repeated in various parts of the tank and the samples mixed. Bottle-sampling from varying portions and depths of the reservoir may be carried out. A single drum of oil may be sampled in the usual way by means of a glass tube.

Gas.—To obtain a sample of the gas from a well, a 1 or 2 quart bottle with glass stopper should be turned bottom upwards and the rubber connection with the pipe from which the gas is issuing passed into the bottle, which is then allowed to remain for fifteen minutes or more. The bottle is closed with a greased stopper, and finally sealed with paraffin-wax. A. R. T.

Analysis of Lactic Acid. W. Klapproth. (*Chem. Zeit.*, 1911, **35**, 1026-1027.)—The following method of titration is recommended for the estimation of lactic acid and lactic anhydride in samples of lactic acid: One grm. of the sample is diluted with 20 c.c. of distilled water, and the solution is titrated with $\frac{N}{1}$ sodium hydroxide solution, phenolphthaleïn being used as the indicator. The number of c.c. of $\frac{N}{1}$ solution required for the titration, when multiplied by 9, gives the percentage weight of the free acid in the sample—that is, the lactic acid *plus* one-half of the lactic anhydride. An excess of 1 c.c. of $\frac{N}{1}$ sodium hydroxide solution is now added to the neutral solution (in the case of samples containing more than 50 per cent. of lactic acid 3 c.c. should be added), and the mixture is heated on a boiling-water-bath for five minutes; it is then titrated with $\frac{N}{1}$ sulphuric acid, an excess of 1 c.c. of the latter is added, the mixture is again heated for two minutes, and titrated with $\frac{N}{1}$ sodium hydroxide solution. The quantity of sodium hydroxide solution used in the two titrations, less the amount of sulphuric acid added, when multiplied by 9, gives the total percentage of acid (lactic acid *plus* anhydride) in the sample, expressed in terms of lactic acid. W. P. S.

Estimation of Minute Quantities of Nitroglycerin. W. L. Scoville. (*Amer. J. Pharm.*, 1911, **83**, 359-364.)—In the author's experience the nitrometer method and saponification with alcoholic alkali are not generally reliable for this estimation, though the Kjeldahl method and the phenol-disulphonic acid method give good results. The latter may be carried out as follows: An amount of alcoholic solution of the substance, equivalent to 0·00065 grm. (or $\frac{1}{100}$ grain) of nitroglycerin, is allowed to evaporate spontaneously. If tablets are to be examined, five $\frac{1}{100}$ grain tablets are powdered, shaken with 10 c.c. of alcohol during one to two hours, and the mixture filtered. Two c.c. of this filtrate are then treated similarly. A solution of potassium nitrate containing the equivalent of 0·0001 grm. nitrogen per 1 c.c. contains, in 1·2 c.c., an amount of nitrogen equal to that present in $\frac{1}{100}$ grain pure nitroglycerin, and 1·2 c.c. of this standard solution is thus evaporated at low temperature; when the standard and sample are dry, 2 c.c. of the phenol-disulphonic acid reagent are added, the mixture stirred well, allowed to stand ten minutes, diluted with water, made slightly alkaline with potassium hydroxide, and diluted to 100 c.c., and the colours compared in the usual way. A. R. T.

Use of Miscibility Curves in the Examination of Essential Oils and Balsams. E. Louïse. (*J. Pharm. Chim.*, 1911, **4**, 193-197.)—Two c.c. of the essential oil under examination are mixed with 2 c.c. of standard petroleum spirit and increasing quantities of aniline from 5 to 30 c.c., and the miscibility curves plotted as described in previous communications (ANALYST, 1907, **32**, 365; 1910, **35**, 322). Lavender oil gives a curve far removed from that of turpentine oil or terpenyl acetate, which is sometimes used to enrich lavender oil poor in esters. The curves given by bigarade neroli oil and petit-grain oil are also distinct, the maximum difference being about 3° C. French, Japanese, and American peppermint oils gave parallel curves sufficiently far apart to enable the three kinds of oil to be distinguished. Similar differences were observed between copaiba balsams of different origin.

C. A. M.

Estimation of the Specific Gravity of Oils. E. Bellmer. (*Chem. Zeit.*, 1911, **35**, 997.)—The method of comparison with mixtures of alcohol and water affords an accurate means of determining the sp. gr. of viscous oils. A few drops of the oil under examination are introduced into 2 c.c. of absolute (98 to 99 per cent.) alcohol, and water added little by little from a burette until the drops just begin to float. The sp. gr. of the dilute alcohol is then the same as that of the oil. The value may be readily found by reference to the following table from the amount of water added:

Water c.c.	Sp. Gr. at 15° C.	Water c.c.	Sp. Gr. at 15° C.	Water. c.c.	Sp. Gr. at 15° C.	Water c.c.	Sp. Gr. at 15° C.
0·1	0·8178	1·1	0·9050	2·1	0·9376	3·1	0·9537
0·2	0·8331	1·2	0·9091	2·2	0·9397	3·2	0·9548
0·3	0·8456	1·3	0·9133	2·3	0·9417	3·3	0·9558
0·4	0·8563	1·4	0·9173	2·4	0·9436	3·4	0·9568
0·5	0·8678	1·5	0·9209	2·5	0·9453	3·5	0·9577
0·6	0·8739	1·6	0·9243	2·6	0·9469	3·6	0·9587
0·7	0·8812	1·7	0·9273	2·7	0·9485	3·7	0·9595
0·8	0·8880	1·8	0·9301	2·8	0·9499	3·8	0·9603
0·9	0·8940	1·9	0·9327	2·9	0·9512	3·9	0·9610
1·0	0·8995	2·0	0·9353	3·0	0·9525	4·0	0·9617

C. A. M.

Estimation of Rosin in Fatty Oils. W. Fahrion. (*Chem. Rev. Fett- u. Harz-Ind.*, 1911, **18**, 239-242.)—About 5 grms. of the oil under examination are dissolved in a mixture of 50 c.c. of petroleum spirit and 20 c.c. of 96 per cent. alcohol, and the solution rendered faintly alkaline to phenolphthaleïn with $\frac{N}{1}$-alkali solution. After the addition of sufficient water to bring the alcoholic strength to about 60 per cent., the liquid is thoroughly shaken, an extra 1 c.c. of $\frac{N}{1}$ alkali solution added, and the alcohol evaporated on the water-bath. The residue is taken up with water, and the fatty and rosin acids liberated by shaking the liquid with several successive portions of ether and hydrochloric acid. The mixed acids left on evaporation of the ether are next esterified by treating them with 20 c.c. of absolute alcohol, adding 20 c.c. of petroleum spirit and 1 c.c. of concentrated hydrochloric acid, shaking the mixture vigorously in a separating funnel, and allowing it to stand overnight. The liquid is again rendered faintly alkaline, the alcohol once more diluted to about 60 per cent. strength, and the shaking repeated. Finally, the last traces of the ethyl esters of the fatty acids are eliminated by shaking the clear rosin soap solution with petroleum spirit, after which the alcohol is evaporated, the rosin acids liberated by treatment with ether and hydrochloric acid, the ethereal solution evaporated on the water-bath, and the residue dried for an hour at 100° C. and weighed. Results obtained in test experiments with linseed oil containing known quantities of rosin were in close agreement with theory, but an estimation in which only rosin was present gave a result 2·55 per cent. too low. From this it would appear that the relative proportion of fatty and rosin acids present had some influence upon the esterification. The proportion of unsaponifiable matter found in linseed oil containing rosin was also too low, and it is therefore recommended either that the average proportion of unsaponifiable matter in linseed oil should be taken as 1 per cent. (instead of 1·5 per cent.), or that the results should be expressed as the proportion of rosin acids found and not calculated into colophony. C. A. M.

Estimation of Fillers (Mineral Matter) in Rubber Mixtures. Brüggemann. (*Gummi Zeit.*, 1911, **25**, 1529-1530; through *Chem. Zentralbl.*, 1911, II., 801.)—The rubber material is extracted with acetone and then boiled with petroleum oil as described by Hinrichsen (Analyst, 1910, **34**, 406). After diluting with benzene, the mixture is allowed to settle, and the insoluble matter is collected on a filter prepared as follows: Two thicknesses of filter-paper are placed in a Gooch crucible, and a mixture of kieselguhr and water is poured into the crucible; before all the water has drained through, alcohol is added, and then ether. The crucible and its contents are next dried and weighed. The same filter may be used again after the surface of the kieselguhr has been removed to a depth of about 0·2 mm.

W. P. S.

The Application of Bromination Methods for the Estimation of Caoutchouc in Vulcanised Products. W. Esch. (*Chem. Zeit.*, 1911, **35**, 971-972.)—The various methods which have been proposed for the estimation of caoutchouc in vulcanised products by the action of bromine, whether in organic solvents or aqueous media, all depend on the following assumptions: (1) That 136 parts of caoutchouc hydrocarbon combine additively with 319·7 parts of bromine or 64 parts of sulphur; (2) that if 136 parts of caoutchouc have already combined with 32 parts of sulphur as the result of vulcanisation, they can only combine further with 159·85 parts of bromine; (3) that the whole of the sulphur combined with the vulcanised caoutchouc is unaffected by the bromine; (4) that the difference between the total weight and the weight of the combined sulphur and bromine represents the weight of caoutchouc; (5) that for every 64 parts of combined sulphur, or 319·7 parts of combined bromine, 136 parts of caoutchouc hydrocarbon may be calculated. The author has made a large number of analyses, from which he concludes that none of the above theoretical assumptions are actually valid. If the brominated sulphur derivative be weighed, and the weights of the combined bromine and sulphur be deducted, the results may be at one time 15 per cent. too low, and at another 23 per cent. too high. On the other hand, if the caoutchouc be calculated according to Hübener's method from the values found for combined sulphur and bromine, the results range from 7 per cent. too low to 24 per cent. too high. Further, the same kind of errors are not always found with the same samples. It is hardly to be expected that the combined sulphur of vulcanisation, which is appreciably susceptible to the oxidising action of the air, should be capable of resisting entirely the action of one of the most powerful oxidising agents known—namely, bromine, or bromine water.

J. F. B.

Estimation of Soap in Heavy Grease. W. Bockermann. (*Chem. Zeit.*, 1911, **35**, 1066.)—The following method is recommended as sufficiently accurate for most technical purposes. After estimation of the free fatty acids, 2 grms. of the grease are boiled for thirty minutes on the water-bath beneath a reflux condenser with 50 c.c. of $\frac{N}{10}$ potassium hydroxide solution and 50 c.c. of absolute alcohol. The contents of the flask are then cooled, shaken with 40 c.c. of petroleum spirit, and allowed to stand until separate layers are formed, after which 20 c.c. of the petroleum spirit layer are withdrawn and evaporated, and the residue of unsaponifiable matter dried on an oil-bath at about 140° C. until constant in weight. The difference between 100 and the percentage result gives the amount of soap in the grease. When a large quantity of free fatty acids is present, a corresponding correction must be made.

C. A. M.

Reaction of Sparteine. A. Jorissen. (*J. Pharm. Chim.*, 1911, **4**, 251-252.)—The ammonium sulphide test for sparteine described in the French *Codex* is rendered more sensitive when applied as follows: If 0·01 grm. of sparteine sulphate is placed on a strip of filter-paper and moistened with a drop of ammonium sulphide (containing polysulphides), a brownish-red coloration is produced as the liquid evaporates. This coloration is manifested in the absence of moisture, and appears to depend largely upon the presence of polysulphides. A still better modification is to add a slight excess of sodium hydroxide to a solution of a trace of sparteine sulphate in the smallest possible quantity of water, and to shake the liquid with about 10 c.c. of ether. The ethereal layer is decanted and shaken for about a minute with 0·01 to 0·02 grm. of dry sulphur, after which a current of hydrogen sulphide is introduced. This soon produces a voluminous red precipitate, which disappears on the addition of water. The reaction is clearly given by 0·01 grm. of sparteine sulphate, whereas neither other alkaloids nor pyridin behave in the same way. Coniine, however, gives an orange-yellow turbidity, whilst atropine yields a yellow precipitate.

C. A. M.

INORGANIC ANALYSIS.

Precipitation of Aluminium, Chromium, and Iron by Ammonium Nitrite. E. Schirm. (*Chem. Zeit.*, 1911, **35**, 979-980.)—In a previous communication (*Chem. Zeit.*, 1909, **33**, 877) it was shown that in presence of more than 2 per cent. of ammonium salts in the solution the precipitation of metallic hydroxide by ammonium nitrite may not take place. If the nitrous fumes be expelled by boiling, and precipitation be then effected by ammonia, the precipitate, though not possessing the granular form of the precipitate formed by the nitrite, is, nevertheless, far more readily filtered than one formed by ammonia alone, owing to the mechanical action of the nitrogen gas evolved. It has been found that the hydroxides of this group may be precipitated by sodium nitrite, regardless of the quantity of ammonium salts; but then the liquid cannot be used for the subsequent estimation of the alkalis. It is suggested that since the nitrites of the organic quaternary ammonium bases react in all respects similarly to the nitrites of alkali metals, one of these—for instance, trimethylphenylammonium nitrite—might get over both difficulties.

The author has attempted to effect the separation of aluminium, chromium, and iron from the diad metals of the ammonium sulphide group by means of the nitrite method of precipitation. Solutions of zinc, cobalt and nickel salts are not precipitated by ammonium nitrite, whilst manganese is partially precipitated in absence of ammonium salts, but not in presence of small quantities of these salts. The separation of iron from manganese is effected by precipitating the ferric hydroxide in the manner previously described, but with the addition, before precipitation, of a little ammonium sulphate. The filtrate is acidified with sulphuric acid, boiled, and the manganese estimated by the persulphate method. The manganese results come out about 2 per cent. too low owing to a slight co-precipitation with the iron, but exact results may be obtained by a double precipitation. If more than 3 grms. of ammonium sulphate be added per 250 c.c., precipitation of the iron is incomplete, and the subsequent use of ammonia is not admissible in this case, since it increases the co-precipitation of manganese. Here also better results are anticipated by the use of trimethylphenylammonium nitrite. Attempts to separate the triad metals from zinc by the nitrite method failed, since a large proportion of the zinc was always carried down in combination with the sesquioxide. J. F. B.

Analysis of Calcium Cyanamide. H. Kappen. (*Chem. Zeit.*, 1911, **35**, 950-952.)—In contradiction to Monnier (Analyst, 1911, 429), and in support of Stutzer (Analyst, *loc. cit.*), the author finds no difference in the results obtained by the ordinary Kjeldahl method and those obtained when the digestion is carried out after addition of salicylic acid and zinc dust, or of salicylic acid and thiosulphate as suggested by Monnier. Nor does moistening the material with water before addition of sulphuric acid affect the final result. He concludes that commercial calcium cyanamide yields all its nitrogen as ammonia when 0·5 grm. is digested with 30 c.c. sulphuric acid (*cf.* Dinslage, following Abstract). It is suggested that Monnier's results would probably be explained if that author had described his method in detail, and in the present paper full detail is given.

For the estimation of the cyanamide nitrogen as distinguished from total nitrogen, the author uses his modification (*Landw. Versuchsstationen*, 1909, **70**, 445) of Perotti's method (*Gaz.*, 1905, **35**, II., 228). This method, which depends on the precipitation of cyanamide by an ammoniacal solution of silver nitrate ($\frac{N}{100}$), and titration of the excess of silver by means of thiocyanate, has been criticised by Caro (Analyst, 1910, **35**, 76) on the ground that the composition of the precipitate is variable, though it contains the whole of the cyanamide. If the author's directions (*loc. cit.*) are rigidly adhered to, the precipitate has the composition indicated by theory. The experiments of Caro (*loc. cit.*), who recommended precipitation by an ammoniacal solution of silver acetate, washing the precipitate and determining its nitrogen content by Kjeldahl's method, appear to have been made exclusively with the commercial product known as "Kalkstickstoff." When the corresponding precipitate of silver cyanamide from "Stickstoffkalk," a commercial product distinguished from "Kalkstickstoff" by containing calcium chloride (and tar products, *cf.* Dinslage, following Abstract), is treated with concentrated sulphuric acid, a violent reaction attended by loss of nitrogen occurs. The nitrogen content of the precipitate may, however, be determined accurately by Kjeldahl's method, if a little water be added before the sulphuric acid.

It is pointed out that Caro's method (*loc. cit.*) for the estimation of dicyandiamide necessarily estimates as such any dicyandiamidine present. G. C. J.

[*Note by Abstractor.*—Caro showed that dicyandiamidine was not present in those brands of calcium cyanamide with which he worked.]

Analysis of Calcium Cyanamide and of Norwegian Nitre. E. Dinslage. (*Chem. Zeit.*, 1911, **35**, 1045-1046.)—*Calcium Cyanamide.*—In the author's experience the only way in which the whole of the nitrogen can be obtained as ammonia is to digest the substance with dilute sulphuric acid (1 : 1). This digestion is carried out in an ordinary 600 c.c. Jena flask (not a Kjeldahl flask) and 1 grm. of the material, 30 c.c. of dilute sulphuric acid, and a drop of mercury are taken. Decomposition is complete in one and a half to two hours. Working in this way, four different chemists found 19·19 to 19·24 per cent. nitrogen. Using concentrated acid at the start, the same sample yielded from 17·22 to 19·23 per cent. (mean of 6, 18·57 per cent.) nitrogen as ammonia. Preliminary moistening of the sample with water or alcohol appears to effect a small improvement, but the results are not concordant, and the mean values so obtained are notably lower than those obtained by the author's method. Nor does the addition of reducing agents effect any measurable improvement (*cf.* Kappen, preceding Abstract), unless at the same time the initial concentration of sulphuric acid is reduced, when the improvement is due to this diminished concentration and not to the reducing agent. The above results were obtained with the commercial product known as "Kalkstickstoff," but similar results were obtained with "Stickstoffkalk," and are given in the paper. "Stickstoffkalk" contains from 5 to 6 per cent. of calcium chloride, and the samples examined by the author yielded to ether between 6 and 7 per cent. of a yellow, unsaponifiable oil with a tarry odour, which odour and its blacker appearance distinguish "Stickstoffkalk" from "Kalkstickstoff"; the latter usually smells faintly of acetylene. The oil, which is presumably added to diminish the dust nuisance which attended the use of calcium cyanamide when first introduced, makes it very difficult to wet this product.

Norwegian Nitre.—The estimation of moisture in Norwegian nitre presents considerable difficulty. When dried alone at 100° to 105° C., the process is incomplete after as long as three weeks. In admixture with dry sand better results can be obtained in three days, but they are still below the truth (19·9 instead of 20 per cent.). The last 0·5 per cent. cannot be removed by exposure to the action of desiccating agents in an exhausted vessel. On the other hand, the determination may be made with great exactness in twenty minutes if the following directions are followed: From 3 to 5 grms. of the substance are heated in a crucible situated some

10 cm. above a small flame. At the end of the process the contents of the crucible should have an irregular form and opaque appearance. If heated too strongly, the mass melts a second time and forms at its edges a compact, transparent cake; this change is attended with loss of matter other than water. The estimation of nitrate and nitrite present no difficulty, and that of iron, aluminium, manganese and magnesium is simplified by the fact that, with the exception of a trace of magnesium, these remain wholly with the insoluble residue. G. C. J.

Practical Value of Calorimetry in Testing Anthracite. S. F. Peckham. (*Chem. Engineer*, 1911, **14**, 351-355.) As the result of experimental work carried out in controlling the coal supplies of the Corporation of New York, the author arrives at the conclusion that, so far as anthracite is concerned, the use of a calorimeter is not justified, since it gives results which are directly proportional to the fixed carbon, and the latter can be determined more expeditiously and more cheaply than the calorific power. He also protests against the false assumption, made in the specifications of the City of New York, that the practical value of coal bears a simple direct ratio to its calorific power. The practical value falls far more rapidly than the calorific power, and may reach zero when the calorific power is still 50 per cent. of that of the best coal (*Cf.* Pope, ANALYST, 1911, 229). G. C. J.

Analysis of Graphite. F. Mayer. (*Chem. Zeit.*, 1911, **35**, 1024-1025.)—The amount of graphitic carbon in graphite may be accurately and conveniently estimated by taking the calorific value of the sample. For this purpose 0·6 grm. of the graphite is mixed with 0·4 grm. of pure benzoic acid, the mixture is pressed into the form of a tablet, and its calorific value is estimated in a Berthelot bomb apparatus. The benzoic acid is added in order to insure complete combustion of the graphite; 1 kilo. of benzoic produces 6,322 calories, and 1 kilo. of graphitic carbon 7,900 calories. A simple calculation gives the quantity of graphitic carbon from the calorific value found. Should the graphite contain sulphur, the result obtained must be corrected for its quantity; the sulphuric acid resulting from the combustion of the sulphur dissolves in the water contained in the bomb, and the solution is titrated. For each c.c. of $\frac{N}{10}$ solution required for the titration 7·06 calories are deducted from the calorific value of the sample. W. P. S.

Estimation of Carbon in Steel by Means of a Calorimetric Bomb. P. Mahler and E. Goutal. (*Compt. Rend.*, 1911, **153**, 549-551.)—The bomb used is a large one of 1,000 c.c. content, and it is neither lined with platinum nor with enamel, but contains a removable porcelain beaker so as to make it possible to conduct the estimation of sulphur simultaneously. The sample (2 to 5 grms.) is contained in a capsule of fire-clay, free from carbonates, and ignition is effected by means of a piece of fine iron wire, through which an electric current is passed when all is ready. Copper is used in place of platinum for the electrodes and support for the capsule. There are two cocks, to one of which a copper tube is attached, reaching nearly to the bottom of the bomb, the whole arrangement being very similar to that of bombs used for determining the calorific power of gases and oils. It is advisable, as a matter of routine, to mix the sample with litharge, to insure complete combustion, but this is only necessary with very hard steels. Combustion takes place under a pressure of no more than 5 to 8 atmospheres, and at its conclusion the gases are passed through a measured quantity of baryta solution of known strength, which is then titrated with oxalic acid. The results obtained with a number of steels of very different character agreed closely with those supplied by the works (Creusot and Imphy) with each sample, except in two cases. In one of these cases the steel was highly silicious (1·3 per cent.), the other contained 5·1 per cent. of chromium, and the makers' numbers for carbon appear to have been wrong, since the authors confirmed their own higher numbers by liberation of the carbon, followed by its dry combustion. G. C.

Rapid Estimation of Ferric Oxide in Cement. A. Golubinzeff. (*Chem Zeit.*, 1911, **35**, 961-962.)—A weighed quantity (2 grms.) of the cement is brought into an Erlenmeyer flask and moistened with 10 c.c. of water. After ten minutes, a further 75 c.c. of water are added, and then gradually, with shaking, 100 c.c. of $\frac{N}{1}$ hydrochloric acid. Shaking is continued until the greater part of the cement is dissolved, and solution (except of any sandy particles) is completed by warming. The solution is reduced by means of sulphur dioxide or hydrogen sulphide, the reducing agent expelled by passing a current of carbon dioxide through the boiling solution, and the solution cooled in an atmosphere of carbon dioxide and titrated with permanganate in presence of 10 c.c. of Reinhardt's solution (manganese sulphate and phosphoric acid). The whole operation occupies only forty-five to sixty minutes, and the method, which allows of the use of larger quantities of material than are usually taken, gives results which are substantially identical with those yielded by the most exact gravimetric methods, and which agree better among themselves than do duplicate gravimetric determinations. For example, the precipitate of ferric oxide and alumina obtained in the ordinary course of analysis, after separation of silica, was fused with bisulphate, and the melt taken up in dilute hydrochloric acid. After addition of tartaric acid and ammonia to this solution, iron was precipitated by means of ammonium sulphide, the iron sulphide dissolved in hydrochloric acid, with the addition of a little nitric acid, and the iron finally precipitated by means of ammonia, the precipitate tested for the presence of silica, and its weight corrected for the amount found. Duplicate determinations gave 2·82 and 2·89 per cent. Ten determinations by the author's method gave results which lay between 2·82 and 2·88 per cent. G. C. J.

Simultaneous Estimation of Ferrocyanides and Thiocyanates. L. Ronnet. (*Ann. Chim. anal. appl.*, 1911, **16**, 336-337.)—An aliquot portion of the solution is strongly acidified with hydrochloric acid, and treated with a slight excess of a hot solution of ferric chloride. The precipitate of Prussian blue and ferric thiocyanate is digested for thirty minutes on the boiling-water-bath, and then separated and washed with boiling water until the washings are neutral and colourless. The filter containing the Prussian blue is transferred to a graduated flask, into which is also introduced a definite volume of 10 per cent. potassium hydroxide solution (sufficient to dissolve the Prussian blue). The flask is now thoroughly shaken and allowed to stand for twelve to sixteen hours, after which its contents are made up to the mark, shaken, and filtered. Finally, an aliquot portion of the filtrate is slightly acidified with 10 per cent. sulphuric acid and titrated with $\frac{N}{10}$ potassium permanganate, 1 c.c. of which is equivalent to 0·0422 grm. of crystalline potassium ferrocyanide, or to 0·02866 grm. of anhydrous Prussian blue. The filtrate from the Prussian blue is made up to definite volume, and an aliquot part decolorised with sodium bisulphite solution (added in slight excess), heated for a few moments on the water-bath, and treated with a slight excess of 10 per cent. copper sulphate solution. After standing for thirty minutes on the water-bath the liquid is filtered, and the precipitate of cuprous thiocyanate washed with boiling water until the washings are free from copper, after which it is dissolved in ammonia solution (1 : 3), and the solution heated and titrated with $\frac{N}{10}$ potassium permanganate solution. One c.c. is equivalent to 0·0016 grm. of potassium thiocyanate. C. A. M.

Preparation of Pure Potassium Ferrocyanide and its Analytical Control. K. Schroeder. (*Zeitsch. anorg. Chem.*, 1911, **72**, 89-99.)—Crystals of potassium ferrocyanide obtained by cooling, with shaking, a hot concentrated solution of the commercial salt, have the composition represented by the formula $K_4Fe(CN)_6 3H_2O$. Statements to the contrary—*e.g.*, that of Müller and Diefenthäler (*Zeitsch. anorg. Chem.*, 1910, **67**, 421)—are based on indirect determinations of the water of crystallisation, which indicated a somewhat lower percentage of water than corresponds to the above formula. Though it is possible, with suitable precautions, to determine the water of crystallisation by loss in weight on drying, anhydrous ferrocyanide is somewhat hygroscopic, and tends to become oxidised when maintained for long periods at temperatures in the neighbourhood of 100° C.

A convenient means of controlling the purity of potassium ferrocyanide is to decompose it by boiling with concentrated sulphuric acid, and then to precipitate the iron by means of nitrosophenylhydroxylamine (ANALYST, 1910, **35**, 327). The decomposition is conveniently effected in a large (500 to 600 c.c.) Erlenmeyer flask, supported on asbestos in an air-bath of such depth that only the upper third of the flask remains outside it. When the evolution of carbon monoxide is ended, the temperature is raised until the acid begins to fume and is maintained for half an hour. For the decomposition of 2 grms. of ferrocyanide, 15 c.c. of sulphuric acid is a suitable quantity. To the cooled solution, water and 15 c.c. of concentrated hydrochloric acid are added; the mixture is warmed to effect complete solution of the ferric sulphate, and then cooled, and the ferric iron is finally precipitated as stated.

The following new volumetric method is described: It depends on the destruction of the ferrocyanogen complex in such a manner that the whole of the iron is obtained as ferrous salt, which is then titrated with permanganate. When a solution of a ferrocyanide is added to a cold saturated solution of mercuric chloride, mercuric ferrocyanide separates as a yellow precipitate. If the ferrocyanide be added in small quantities to a hot solution of mercuric chloride, the ferrocyanogen complex is completely broken down, and the solution gives the reactions of ferrous iron.

If the addition of ferrocyanide be continued, the speed of its decomposition slackens and ferrous ferrocyanide has a more than momentary existence, and this compound readily undergoes oxidation. The method, therefore, is as follows: The solution of ferrocyanide (2 grms. in 50 to 100 c.c. cold water) is added to 100 c.c. of a cold saturated solution of mercuric chloride. This mixture is added in small portions at a time to 200 c.c. of a boiling solution of mercuric chloride to which 2 or 3 drops of hydrochloric acid (1 : 1) have been added, making fresh additions as fast as the precipitate of mercuric ferrocyanide disappears. Finally, the first flask is rinsed with water, and the washings transferred to the second flask, which should be a large one (1,000 c.c.). The operation takes about fifteen minutes. The cooled mixture is then titrated with permanganate after addition of 30 c.c. of Reinhardt's solution (manganese sulphate and phosphoric acid). The results are strictly concordant, and, on 2 grms. of pure ferrocyanide, are 0·1 per cent. higher than theory, which corresponds to a consumption of 0·05 c.c. more permanganate than is required by theory. G. C. J.

Electro Analysis. (*Report of the Committee British Association,* 1911.)—Attention has been directed during the past year particularly to the application of the electrometric method to the titration of weak acids in such liquids as tan liquors. It was found that the potentiometer-box and auxiliary electrode constructed for the separation of metals by graded potential may be conveniently employed for this purpose in conjunction with a form of hydrogen electrode specially designed to combine ease of manipulation and rapidity of saturation. The question regarding the "end-point" of the titration has been examined. It is known that if the object of the titration be to determine the number of equivalents of acid present, then not only the nature of the acid or acids must be considered, but also the concentration of the salts resulting from the titration. It has been pointed out, however, that in most practical cases it will be possible to fix end-points of special importance for the particular purpose in question. Frequently the liquid may be titrated until the hydrion-concentration of pure water is reached. This corresponds to a potential difference between the hydrogen-electrode and the normal calomel electrode recommended for these titrations of 0·69 volt (ANALYST, 1911, 121).

It has been found possible by Hildebrand and E. F. Smith and his co-workers (ANALYST, 1907, **32**, 268) to electrolyse the alkali metals with a mercury cathode and to analyse both the anion and cation. For this purpose a double cell is employed, the inner and outer portions being sealed by means of mercury. The alkali salt to be analysed, say potassium sulphate, is placed in solution in the central cell, where an anode of platinum gauze is rapidly rotated. The outer cell contains water, with a small quantity of sodium chloride solution to make it conductive. On electrolysing the SO_4 anions are discharged at the anode, and a solution of sulphuric acid obtained. The K cations are discharged on the mercury, which is made the cathode. Owing to the rotation of the anode, the amalgam, which is specifically lighter than the pure mercury, is swept into the outer compartment, where it is decomposed by means of an auxiliary nickel cathode placed above the mercury in the solution. When the electrolysis is complete, the SO_4 in the inner cell is titrated by means of standard alkali, and the K in the outer cell by means of standard acid.

There are disadvantages in the apparatus of Hildebrand, the outer cell of which is a glass crystallising basin, the inner consisting of a beaker with the bottom cut off. The inner cell is kept in a central position by having corks wedged at the sides. F. M. Perkin has now had a vessel made of fused quartz. The centre cell is kept concentric by having quartz rods fused to it and to the inside wall of the outer basin. Being of quartz, the vessel can, for cleaning purposes, be heated to redness, is absolutely unattacked by alkali, and there are no corks which, if they get accidentally splashed, absorb some of the alkali and vitiate the results.

Experiments on the analysis of the anions and cations are being carried out with this apparatus, and the results so far have been encouraging.

Estimation of Manganese as Oxide. P. N. Raikow and P. Tischkow. (*Chem. Zeit.*, 1911, **35**, 1013-1015.)—Having regard to the doubt which has been cast on methods which depend on the weighing of manganese as one of its oxides, a doubt which found its strongest expression in a paper by Gooch and Austin (*Zeitsch. anal. Chem.*, 1899, **38**, 690), the authors have made a study of the conditions which determine the conversion of one oxide into another. Mn_3O_4 or Mn_2O_3 can be reduced quantitatively to MnO by a few minutes' heating over a Teclu burner in a Rose crucible, through which a brisk current of hydrogen is passed. If the current of hydrogen is insufficient, the reduction may take an hour or more.

Mn_2O_3 is reduced quantitatively to Mn_3O_4 by five minutes' heating over the blowpipe flame in a Rose crucible, through which a current of carbon dioxide is passed. As the carbon dioxide takes no part in the reaction, but serves merely by reducing the partial pressure of the oxygen, the current need not be a brisk one, and should not be, for fear of mechanical loss. MnO can be oxidised to Mn_3O_4 by prolonged heating over the blowpipe flame in an uncovered crucible, but the most convenient means of converting MnO into Mn_3O_4 is to heat the former in an open crucible over a Teclu burner, and then to reduce the resulting mixture of Mn_2O_3 and Mn_3O_4 by five minutes' heating over the blowpipe flame in an atmosphere of carbon dioxide.

Lower oxides can be oxidised quantitatively to Mn_2O_3 by heating to a low red heat over an ordinary Bunsen burner in a Rose crucible through which a current of oxygen is passed.

If the manganese is obtained as carbonate, this can be oxidised to Mn_2O_3 in fifteen minutes in an atmosphere of oxygen, using at first the full heat of an ordinary Bunsen burner, and subsequently reducing the temperature to a low red heat. The Mn_2O_3 may then be reduced to MnO, and a check obtained on the first result.

If it be desired to convert manganese sulphate into an oxide for weighing, the most convenient method is to heat it several times alternately in an atmosphere of hydrogen and in an uncovered crucible. The sulphate is reduced by the hydrogen to sulphite and partially to sulphide, which, on heating in air, give rise to sulphur dioxide and oxides of manganese. When sulphur dioxide is no longer evolved, the mixed oxides are raised to a low red heat in a current of oxygen, and converted wholly into Mn_2O_3. In this way the sulphate may be converted into Mn_2O_3 in twenty minutes.

G. C. J.

Action of Salicylic Acid on Metallic Acids. J. H. Müller. (*J. Amer. Chem. Soc.*, 1911, **33**, 1506-1510.)—Titanium dioxide may be separated from the oxides of columbium, tantalum, zirconium, and thorium as follows: The mixed oxides (0·5 grm. or more) are fused with sodium carbonate, the melt is taken up in 350 to 400 c.c. water, salicylic acid (14 to 15 grms.) is added, and the solution kept at the boiling-point for three or four hours. The titanium remains in solution, the other metals are precipitated as salicylates, which are filtered rapidly, washed with hot water, ignited and weighed as oxides. The precipitate tends to be contaminated with alkali salicylate, and it is recommended that the ignited precipitate be fused with bisulphate, and then treated in the usual manner. Extreme error for columbium, ±0·5 mgrm. on 0·3 grm. taken; for tantalum, +0·8 mgrm. (never negative) on 0·5 grm.; for thorium, +0·5 grm. (never negative) on 1 grm.; and for zirconium, −0·5 mgrm. (never positive) on 0·5 grm. Titanium is precipitated in the concentrated filtrate by means of ammonium hydroxide. A bisulphate fusion is even more necessary in this case. Error for titanium, ±1·3 mgrm. Alkali fluorides prevent the precipitation of columbium and tantalum by salicylic acid.

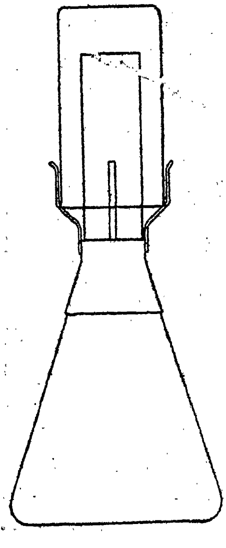
The intense colour produced by the addition of salicylic acid to alkaline titanates in absence of fluorides is recommended as the basis of a colorimetric method for the very accurate determination of very pure titanic oxide, and for the detection of this oxide in acid earths. The sensitiveness of the reaction is great, so little as 0·05 mgrm. in 100 c.c. showing a distinct yellow colour; but its applicability is strictly limited. Not only iron, but zirconium, columbium, and molybdenum, interfere with its quantitative application. As a qualitative test it is scarcely interfered with by zirconium and columbium, as the brown coloration yielded by these elements with salicylic acid is negligible, except when they are present in large quantity, and is distinguished from the yellow colour of titanium solutions by not being discharged by ammonia.
G. C. J.

Use of Sulphur Monochloride in the Determination and Analysis of Rare Earth Minerals. W. B. Hicks. (*J. Amer. Chem. Soc.*, 1911, **33**, 1492-1496.)—Columbium, tantalum, titanium, and tungsten may be readily and completely separated from the rare earth metals by subjecting the finely ground mineral, contained in a boat in a heated combustion-tube, to the vapours of sulphur monochloride. At the end of the operation dry hydrogen chloride is passed through the tube, and the volatile chlorides are finally driven out of this by the use of a Bunsen burner held in the hand. The acid earth metals and most of the iron are volatilised as chlorides, and, together with the excess of sulphur monochloride, pass into the receiver, which is charged with dilute nitric acid. The lumps of sulphur which separate in the receiver are broken up, and the sulphur is dissolved by addition of ammonia and ammonium sulphide. The solution which contains all the tungsten is filtered from the oxides of titanium, columbium, tantalum, and iron, and the tungsten sulphide in the filtrate is precipitated by addition of acid, is washed with water, alcohol, and carbon disulphide, oxidised by means of nitric acid, ignited, and weighed as WO_3. The mixture of acid earths with ferric hydroxide, as well as the non-volatile residue in the boat, are then further examined in any convenient manner.

The above method has been used in opening up large quantities (50-100 grms.) of rare earth minerals which were specially examined for the presence of scandium, which would remain with the non-volatile portion. Neither fergusonite, eschynite, euxenite, nor samarskite yielded even spectroscopic traces of scandium. On the other hand, it is shown that fergusonite may contain 2 or 3 per cent. of thorium, a metal not generally mentioned as a constituent of this mineral. G. C. J.

Rapid Qualitative Detection of Elements, the Sulphides of which are Precipitated by Hydrogen Sulphide in Acid Solution. E. Pozzi-Escot. (*Bull. Soc. Chim.*, 1911, [iv.], 9, 812-814.)—A rapid method for separating the sulphides of the metals of the hydrogen sulphide group, where a quantitative separation is not required, has been worked out by the author. The precipitated sulphides are treated with hydrochloric acid (1 : 4); the sulphides of lead, antimony, tin, cadmium and zinc go into solution, whilst the sulphides of copper, molybdenum, arsenic, bismuth, mercury, gold, and platinum remain insoluble. The solution of sulphides is treated with ammonia, which gives a precipitate with lead, antimony, and tin, whilst cadmium and zinc are not precipitated. The metals are then identified by known tests. The sulphides which resist treatment with hydrochloric acid are treated with nitric acid. Those of copper, molybdenum, arsenic, and bismuth go into solution, and the solution is employed for the identification tests. The sulphides unattacked by nitric acid are dissolved in aqua regia, and the nitric acid is expelled by treatment with hydrochloric acid; the solution is then tested for the presence of mercury, gold, and platinum. J. F. B.

APPARATUS, ETC.



Flask for Quantitative Fermentation Experiments. H. Franzen. (*Chem. Zeit.*, 1911, 35, 987.)—In quantitative fermentation experiments it is necessary, among other things, to provide for a uniform exchange of air. According to the supply of air to the culture, the conditions under which the bacteria develop are modified. In comparative experiments it is particularly necessary that the bacteria should always have the same available supplies of oxygen. The ordinary cotton-wool plugs in no way provide for this uniform circulation of air, but the flask illustrated here has been specially designed for this purpose. It consists of an Erlenmeyer flask with a long neck, the edges of which are not turned over in the usual way. The flask carries a metal collar with wire clips, on which an inverted glass cap is supported. Nutrient solutions kept in these flasks, protected from unnecessary draughts, may be relied on to remain free from germs for at least ten days; an infection by bacteria has not been observed in any case. J. F. B.

Apparatus for the Estimation of the Melting-Point of Fats. L. von Liebermann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 294-295.)—A U-tube is partially filled with mercury, and into one of the limbs a quantity of the molten fat is poured so as to form a layer above the mercury. A thermometer and a platinum wire are fitted into the tube so that their ends are immersed in the layer of fat. When the latter has solidified, a quantity of mercury is poured on its surface, a second platinum wire is placed in the other limb of the U-tube, and the two wires are then connected with an electric bell and battery. The U-tube is now placed in a water-bath, and the temperature is raised gradually; when the fat begins to melt, the mercury above it passes downwards, making connection between the two platinum wires through the main volume of mercury, and the bell rings.

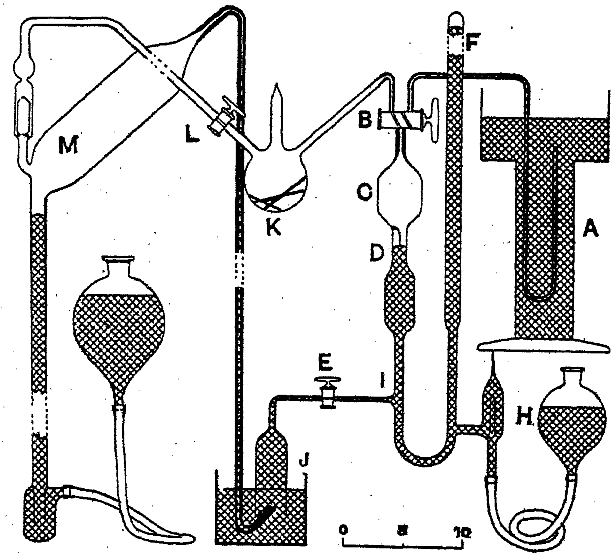
W. P. S.

Use of Indicators for determining Affinity Values. V. H. Veley. (*British Association*, 1911.)—A brief account was given of the use of indicators, especially methyl orange, for determining the affinity value of acids, as also of bases by the hydrolysis values of their hydrochlorides. The advantages, limitations, and defects of the method proposed were discussed in detail, and especial stress was laid upon the fact that the method, though simple in its execution, is not intended to supersede other methods of greater accuracy, available under like conditions. The data of the hydrolysis, and affinity values deduced therefrom, are independent of the more or less conflicting theories—namely, the ionic and chromophoric—of indicators. Exception was more particularly taken to any correction of the experimental results for the hydrogen ion concentration of the solutions, such correction being based upon theoretical speculations based upon the former theory, which has not met with universal acceptance.

Combined Fixing and Staining Processes for Microscopical Preparations. F. Strecker. (*Zeitsch. wiss. Mikroscop.*, 1911, **28**, 17-21; through *Chem. Zentralbl.*, 1911, II., 986-987.)—For fixing and staining sections of kidneys and other organs the author recommends a mixture of 10 or 20 per cent. formaldehyde solution with an equal volume of Ehrlich-Blondi's triacid reagent. A mixture consisting of 3 parts of toluidine blue dissolved in 100 parts of 10 per cent. formaldehyde solution may also be used, followed by treatment with ammonium molybdate.

W. P. S.

Volumetric Estimation of Oxygen in Air. H. E. Watson. (*J. Chem. Soc.*, 1911, **99**, 1460-1466.)—The apparatus used by the author is shown in the accompanying figure. The air to be tested is collected in test-tubes of just over 50 c.c. capacity, on the top of which some potassium hydroxide has previously been fused. The tubes are inverted over mercury, and the gas allowed to remain for two hours to remove carbon dioxide and water-vapour. The gas is admitted into the burette C through the inverted syphon A and the two-way tap B, but some small volume of gas is left in the syphon to avoid filling the tube between B and C with mercury. By means of the reservoir H the height of the mercury is adjusted so that it just reaches a point, D, which is set in the middle of a piece of tubing 1 cm. in diameter, and exactly similar to the manometer-tubing F. F may be either evacuated, boiled out, and sealed off, or preferably connected with a small pump maintaining a good vacuum. The difference in mercury level in F and at D is read off by cathetometer, and the meniscus heights noted. A water-jacket (not shown in figure) completely surrounds the burette and manometer-tube, the water being at room temperature, and kept uniform to $\frac{1}{50}$° C. previous to and during the measurements. The thin glass bulb K, of 60 c.c. capacity, contains 5 grms. of yellow phosphorus, introduced through the tube at the top of the bulb, this tube being afterwards sealed off, and K evacuated by means of the Antropoff pump M, with a delivery-tube of not more than 0·75 mm. diameter. The air passes into K after measurement (L being closed), the mercury in C being passed well beyond the tap B, which is then turned off. K is now heated with a smoky flame for about five minutes, during which time the oxygen is completely absorbed.



After twenty minutes' standing, when the vapours have settled, B is opened and H lowered till the mercury falls just below the connection I, and half the gas passes into the burette. B is closed, and the remaining gas pumped into J, which has a capacity of 20 c.c., and is completely filled. The delivery-tube of the pump must be tapped after the pumping in of the gas, to disengage the final bubble remaining on the end of the tube. E is opened, and the gas from J passes into the burette C. The pressure in C is sufficiently low to cause the mercury to rise in J and pass to I. The tube between these points is of 0·5 mm. bore, and carefully blown on at each end, so that no bubbles of air can be caught in it. The tap E should be turned completely round once or twice to insure that no bubble remains. H is raised carefully while the mercury is running from I, and if any air remains at I, it is displaced by again lowering H. The mercury is now raised to point D, the temperature and pressure measured after a short interval, and the gas expelled through A, the complete analysis being made in one and a half hours, allowing fifteen minutes between each pressure reading and for the complete evacuation with the pump. The phosphorus may be used many times over. It is essential before starting work for the first time that the dry nitrogen formed in the first combustion be circulated through all parts of the apparatus several times. If p be the pressure of the air, T its absolute temperature, and $p'T'$ the corresponding quantities of gases remaining after absorption, the percentage of these gases is given by $\frac{100\,p'T}{pT'}$. The total maximum error of the method does not exceed 6 parts in 15,000.

A. R. T.

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GOVERNMENT REPORTS.

Report of the Principal Chemist upon the Work of the Government Laboratory for the Year ending March 31, 1911, with Appendices. (*Issued as a Parliamentary Paper*, Pp. 1-27.)

Work in connection with the Board of Customs and Excise, Government Departments, the Food and Drugs Acts, and the Fertilisers and Feeding-Stuffs Acts, entailed the analysis and examination of 186,044 samples during the year, against 170,033 for the preceding year.

Five hundred and fifteen samples of beer, out of a total of 7,217 samples taken from retailers, were found to be adulterated with water, and small quantities of saccharin were detected in some cases. Of 638 samples of beer, wort, and materials used by brewers, 41 samples were found to contain arsenic in excess of the limits laid down by the Royal Commission on Arsenical Poisoning; 6 samples of malt, out of a total of 83, contained arsenic in quantities varying from $\frac{1}{90}$ to $\frac{1}{15}$ of a grain per pound. Of 171 samples of glucose, invert sugar, and caramel, none contained arsenic in excess of the limit. One hundred and seven samples of mineral acids, chemicals, drugs, vinegar, etc., were also examined as to the presence of arsenic.

Of 1,398 samples of imported butter examined in accordance with the provisions of the Sale of Food and Drugs Act, 1899, and the Butter and Margarine Act, 1907, 482 samples were found to contain boron preservative, 93 added colouring matter, and 4 an excess of water. One sample of milk-blended butter was imported in packages not marked as required by statute, and proceedings were taken against the importer. Four hundred and eighty-one samples of imported margarine were free from excess of butter-fat, but 7 of these samples contained water in excess of the legal limit. One sample of sweetened condensed milk, out of a total of 83 samples, was found to be prepared from milk which had been deprived of more than half its fat; all the samples of imported milk examined were free from preservatives and from added colouring matters. The amount of fat present in imported creams varied from 11·2 to 56·2 per cent.; 40 out of 58 samples of French cream contained boron preservatives, and calcium sucrate was present in 4 of the samples. Two hundred and thirteen samples of sheep-dips were submitted to the Board of Agriculture and Fisheries; 34 of these samples were deficient in active ingredients at the dilution recommended by the manufacturers, and in 92 other cases the formulæ required modification. The miscellaneous samples examined for this Board numbered 261, and comprised butter, milk, milk-powder, cheese, cider, rice, meals, seeds, feeding-stuffs, soils, fertilisers, poisoned fish, and river water.

The number of samples referred by magistrates under the provisions of the Food and Drugs Act was 83; one sample of milk was not examined on account of leakage from the bottle during transit. In the case of 9 of the samples, the reports of the public analysts could not be confirmed; two of these reports related to milk. Five samples of butter were reported by the public analyst to contain excess of water, 3 to contain foreign fat, and 1 to contain boric acid; the reports could not be confirmed in two cases of alleged excess of water and in two cases where foreign fat was stated to be present. One sample of lard was alleged to contain cocoanut oil, and another "animal membrane" foreign to lard, but these allegations could not be supported. Apple pulp was found to be present in a sample of black currant jam, as reported by the public analyst. The Government laboratory was unable to agree with the analyst in a case in which malt vinegar was alleged to contain artificial vinegar.

The number of samples examined under the Fertilisers and Feeding-Stuffs Act was 20. The samples comprised among fertilisers, superphosphates, dissolved bones, kainit, and various compound fertilisers; among feeding-stuffs, cotton and linseed cakes, sharps, fish-meal, and special feeding meals. The allegation made against the manures was that they were deficient in the guaranteed proportions of phosphate, nitrogen, or potash, and with regard to the feeding-stuffs that they were deficient in albuminoids, and that they contained some worthless or deleterious ingredient.

W. P. S.

Report to the Local Government Board on an Inquiry as to Condensed Milks; with Special Reference to their Use as Infants' Foods. [*Food Reports*, No. 15.] **F. J. H. Coutts.**—Emanating from the Medical Department of the Local Government Board, this report treats of condensed milk chiefly from the point of view of the medical officer, but contains a good deal of information that may prove useful to the analyst. Unfortunately, the chemical portions of the report are neither so full nor so up-to-date as would doubtless have been the case had the report been more, comprehensive; thus, quite a number of papers giving the composition of condensed milks published in the ANALYST have been overlooked, a considerable number of the analyses quoted are incomplete, and, of the detailed analyses, but few have been made within the last fifteen years. The composition of condensed milks is thus stated in terms more general than precise, and the author of the report has been led to overestimate the general degree of condensation, which, although he does not state it in very precise terms, he takes as one-third (pp. 17 and 40), while the analytical figures, as well as a general experience, show a proportion approximating more nearly to 1 to 2¾ in the case of full-cream sweetened milks, and rather less in the case of unsweetened milks.

A point, which may escape observation, though it is not ignored in the report, is the magnitude of the condensed milk industry in the United Kingdom; no analyses are given of British condensed milks, though in the case of three brands it is stated that their origin is Great Britain or a foreign country; the statistics given refer entirely to imports from abroad, and the wording of the paragraph devoted to the manufacture in the United Kingdom is so framed, doubtless unintentionally, as to give the impression that the industry is but a small one.

It is pointed out in the report that no samples of condensed milk examined are free from bacteria, though it appears to be proved that tubercle bacilli, and probably other pathogenic organisms do not survive the process of condensation; as to the origin of the bacteria present, the statements in the report are somewhat conflicting; while it is stated in the report that it is probable that the larger portion of the bacteria found in condensed milk have gained admission during the process of cooling and of filling the tins, in an Appendix, Drs. Gordon and Elmslie appear to come to an opposite conclusion in the case of sweetened milks. It is difficult, however, to agree with the conclusions of the authors of the Appendix, that, in the process of condensing unsweetened milk, sterility is secured, and that the organisms found in these milks are subsequently introduced from the air; as the process of preparing condensed unsweetened milks differs chiefly from that of condensing sweetened milks in that the tins of unsweetened milk are sterilised by heat after condensation and closure, it is not easy to see how the aerial contamination can occur, and this fact casts doubt upon the train of reasoning pursued.

The remarks on labelling will be of interest to Public Analysts, who are doubtless aware of the misleading character of many of the labels, which, though they comply with the regulations of the Sale of Food and Drugs Acts, do not disclose to the casual purchaser the nature of the contents; the unwisdom of coining, by Act of Parliament, a special name, which was previously unknown is brought out by the evidence adduced in the report that "Machine-Skimmed" is taken by many purchasers to indicate a specially high quality, instead of an extreme degree of impoverishment.

The conclusions of the Report are excellent, and, if carried into law, would doubtless tend to prevent many of the evils admittedly connected with the indiscriminate use of condensed milks for infant feeding; these are, briefly:

1. That condensed skim milk should be labelled in a prominent place, and entirely separate, "SKIMMED MILK—UNFIT FOR INFANTS."

2. That the use of any preservative, except sugar, should be prohibited in condensed milk.

3. That a control over false or misleading statements on labels and show-cards should be exercised.

4. That the minimum content of milk-fat, and the maximum of added sugar, should be stated on the label.

5. That the name of the responsible manufacturer or dealer should appear on the tin.

H. DROOP RICHMOND.

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REVIEWS.

THE SOIL SOLUTION: THE NUTRIENT MEDIUM FOR PLANT-GROWTH. By FRANK K. CAMERON. *Bureau of Soils: U.S. Department of Agriculture.* The Chemical Publishing Co., Easton-Penna, 1911. Pp. 136. Price $1·25.

In this work are summarised the views held by Whitney and his colleague, the author, on the nutrition of plants through the medium of the soil. These are in direct contradiction to the views so long held in this country and elsewhere, as the outcome of field experiments such as those of Rothamsted and elsewhere, and which point to the importance of the mineral ingredients in soil and the influence of fertilisers containing them. The author criticises the present methods of soil analysis, and the importance to be attached to this, maintaining that neither the contents of a soil with respect to potash, phosphoric acid, and other mineral plant-food, nor the determination of "available" food by the use of different solvents bear any necessary relation to the crop-producing power of the soil. Ash analyses, he points out, have failed to act as a guide to the fertiliser requirements of a crop.

In place of attaching importance to the mineral constituents, Cameron puts forward two contentions: (1) That the soil problem is one of "dynamic" nature and depends upon the "soil solution," by which is meant the solution formed by the action of the soil-water on the soil particles themselves; (2) that "toxic" substances are formed in the soil as the result of crop-growing, and that the action of fertilisers is to destroy these toxic substances.

The solid components of the soil are always in motion, and it is the movement of the "film water" over the surface of the soil grains which requires to be studied in order to follow a rational practice of soil-control. The statement is made that practically every soil contains all the rock-forming minerals, and hence, but for certain disturbing factors, the concentration of the soil solution is practically the same in all cases, and does not need to be influenced by the presence of further mineral constituents in the soil or the additional supply of them in fertilisers. A description is given of the attempts made to extract the solution naturally existing in the soil and to analyse it. Small quantities were obtained by using powerful centrifuges on saturated soils, and the results seemed to indicate that the concentration of the soil solutions thus obtained differed little with soils of very different textures, and the variations could in no way be correlated with the respective crop-producing powers of the soils. A more ready, if less satisfactory, way of pursuing the inquiry, was found by making an aqueous extract of the soil and passing it through a Pasteur-Chamberland filter, subsequently using the solution in water-culture experiments. As the result, it is stated that there were no recognisable differences in the soluble mineral constituents of good and bad soils respectively, but that the processes going on in the soil tend to form and maintain a normal concentration of the mineral constituents in the soil solution. It is further pointed out that the rise of the capillary water is, in itself, capable of maintaining a sufficient supply of mineral nutrients for crop requirements.

Undoubtedly, it is the case that sufficient attention has not been given in the past to the movements that go on in the soil, and that there is much to learn in this respect, so that the author has done good service in drawing attention to the dynamical considerations that affect crop-growing. But, on the other hand, until some more satisfactory way of getting to know what the "soil solution" really is, one would hardly feel disposed to accept the author's conclusions unreservedly. Moreover, the experimental data on which the conclusions are based are very meagre, and the whole takes the form rather of statements than of proofs. Further, the fact that the conclusions have been based entirely on results obtained in water-culture must tell much against their ready acceptance, for it by no means follows that what takes place under conditions of water-culture would hold equally well in the case of a plant growing in a soil under natural conditions. In face of the long-continued field-work carried on at Rothamsted and elsewhere, as well as of practical experience of the influence of such mineral constitents as potash, phosphoric acid, lime, etc., one may well hesitate to accept, without further proof than can be obtained from soil extracts used in water-culture experiments, the conclusions arrived at as to the practical unimportance of the mineral ingredients in soils.

In the second part of the work the organic constituents of the soil-solution are dealt with, and, once again, soil-extracts have been used, and the results from water-culture have been relied on. Confined as these have been to periods varying from seven to at most thirteen days, the application to actual soil-cultivation conditions must remain very doubtful. The author, however, shows that the soil-solution contains "toxic" substances, several of which have been isolated, and that when a fertiliser has been found to benefit a particular crop growing in a soil, it has also been found to benefit the soil-extract, and to destroy the influence of a "toxic" substance which had proved inimical to a plant growing in water-culture. The differences between extracts from soils known as "good" and those classed as "bad," are stated to be due, not to any differences in their mineral constituents (for these were found to be practically identical) but to the presence of something "toxic" to plant growth. To this, and not to any lack of mineral ingredients, is, in the author's opinion, due the apparent lack of fertility of certain soils. In conclusion, the author comments on the enormous annual waste that has been going on in the misapplication of fertilisers, and urges that the practical problem for the future is, not the seeking of new fertilisers, but the rational use of those at hand. J. A. VOELCKER.

HUILES MINERALES (PÉTROLES, BENZOLS, BRAIS, PARAFFINES, VASELINES, OZOKÉRITE). BY HENRI DELEHAYE. Librarie Polytechnique Ch., Béranger, Paris, 15, Rue des Saintes Pères. 1911. Pp. 215. Price 4 francs.

This useful volume, which forms a part of the Collection des Manuels Pratiques D'Analyses Chimiques, published under the direction of Messrs. F. Bordas and Eugène Roux, may be particularly recommended to those who are interested in petroleum and its products, but are not fully conversant with commercial usage or with the general technical methods of testing. The work deals also, though somewhat more briefly, with the distillates from shale and lignite, in addition to the matters covered by its title. It contains excellent descriptions both of the physical and analytical methods commonly employed for commercial testing work, and includes definitions and technical descriptions of the principal original and finished products, together with tabulated information regarding the most typical, and their constants. A valuable feature is an appendix covering the laws and regulations relating to the petroleum industry in France. The work will be found extremely useful as an adjunct to the possibly more complete, but less technical, works which form the recognised authorities on this subject. Written by one who is especially interested in the practical application of the materials dealt with, the tests recommended for application prior to the distillation and other more elaborate analyses, and the sections dealing with the interpretation of results, and the possibility of determining the source of an oil, are of especial value and interest. Owing to the Customs duties prevailing in France, it is scarcely profitable to import other than the crude oil or partially refined products, and the author considers the subject largely from this point of view, and has thereby broken ground left almost untouched by many other works on petroleum. Unfortunately, although there is a well-arranged table of contents, no index has been supplied with the work.

G. T. Holloway.

Collective Index of the Journal of the Institute of Brewing, 1887-1910. Compiled by W. H. Bird. London: Harrison and Sons, 1911. Price 10s. 6d.

Since 1895 the *Journal of the Institute of Brewing* has formed a comprehensive record of scientific and practical work carried out in connection with brewing, malting, distilling, and the allied industries in all parts of the world. The Collective Index, which is an exhaustive book of reference to the *Journal*, and thus to the entire literature of the fermentation industries, covers 550 pages. Divided into four parts—(1) Authors, (2) Subjects, (3) General Matter, and (4) List of Journals from which Papers have been abstracted—it has been made, by this means and by a system of cross-indexing, almost impossible to miss any reference having a bearing upon the subject to be traced. Part (4) probably represents the most complete list of journals of interest to the fermentation industries extant. Not only will the Collective Index be welcomed by all who have the *Journal* of the Institute and are actually engaged in the industries with which it deals, but the publication itself will be found most useful by everyone who indirectly has an interest in the technology of those industries. Instead of the double columns adopted in many other indexes, the matter is printed exceptionally clearly across the page, and the whole arrangement both of matter and printing greatly facilitates the work of reference.

DECEMBER, 1911. Vol. XXXVI., No. 429.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

NOTE ON THE EXAMINATION OF FINNISH TURPENTINE.

By L. MYDDELTON NASH, F.I.C.

(Read at the Meeting, November 1, 1911.)

The examination of some Finnish turpentine, recently imported, showed that it was normal in all respects except that the close flash was 67° F. instead of from 90° to 100° F. Finnish turpentine gives the Conradsen reaction (a yellow colour on shaking with sulphurous acid, sometimes erroneously referred to as the Hertzfeld reaction), showing that it is distilled from the wood. It was at first thought that the low flash might be due to the presence of a small amount of acetone produced during the distillation of the wood, and, consequently, the three following methods of separation were carried out:

(1) Distillation, using a Young's disc and rod stillhead; (2) shaking out with a strong solution of sodium bisulphite, distilling the bisulphite with carbonate of soda solution, fractionating the distillate by further distillation, and dehydrating with calcium chloride; (3) shaking out with water, fractionating and dehydrating as in (2).

By all three methods a small quantity of "spirit" was obtained, which inflamed at ordinary temperature, and in each case it was found that the turpentine, after separation of the spirit, flashed between 90° and 100° F.

To ascertain whether such a small quantity as seemed to be present was capable of lowering the flash to so great an extent, a mixture of 1 part by volume of acetone and 99 parts by volume of turpentine was made. At 73° F. it flashed with explosive violence. One half per cent. of acetone in turpentine was then tried, and the flash-point found to be 71° F.

A large quantity of the turpentine was then shaken out in successive portions with 100 c.c. of water, the aqueous extract distilled, and the first 25 c.c. collected. This was dehydrated, first with calcium chloride, and then with anhydrous copper sulphate, until, finally, about 2 c.c. of spirit was obtained, which inflamed at the ordinary temperature, burning with a non-luminous flame. On oxidation with bichromate of potassium and sulphuric acid, the pungent odour of formic acid was noticed, and the distillate reduced ammoniacal silver nitrate. The presence of methyl alcohol was thus suspected.

A larger quantity of the turpentine was then shaken out with water which was afterwards fractionally distilled. The distillate was distilled three times over quicklime, and four times over anhydrous copper sulphate, when the sp. gr. at 15·5° C. of the resulting spirit was found to be 0·810. It may be mentioned here that the spirit dissolved some of the copper sulphate, giving a blue solution; methyl alcohol behaves similarly. The spirit obtained was then fractionated in a Wurtz flask and collected in three portions—viz., from 65° to 70° C., from 70° to 75° C., and from 75° to 80° C. Some commercial methyl alcohol of sp. gr. 0·821 was also fractionated, the range of temperature being the same as that of the spirit under examination.

The refractive indices of the two lighter fractions of the spirit were found to be almost identical with those of the two lighter fractions of the commercial methyl alcohol. The portion boiling between 65° and 75° C was further fractionated, using a Young's disc and rod stillhead, and the fraction distilling between 65° and 67° C. was separated for the determination of its vapour density. The molecular weight was thus found to be 31, methyl alcohol being 32. The crystalline oxalate was prepared, and found to have the same appearance and melting-point as that prepared from methyl alcohol. The behaviour of the spirit with barium oxide and with calcium chloride was also characteristic of methyl alcohol. It was therefore evident that the spirit washed out of the turpentine was crude wood alcohol, the methyl alcohol itself being identified.

It may be objected that methyl alcohol is insoluble in turpentine, but, curiously enough, thes pirit obtained was also found to be insoluble in turpentine, consequently, it was impossible to determine the flash-point of a mixture of the two.

In all probability several other substances are present in minute quantities which hold the methyl alcohol in solution in the turpentine.

This note is recorded chiefly to warn those inexperienced in the examination of turpentine against the danger of reporting as necessarily adulterated with light petroleum spirit or benzene, turpentine flashing at ordinary temperature, and yielding by Armstrong's process about 10 per cent. of unpolymerisable residue. This quantity seems to be normal for Finnish turpentine, and is chiefly cymene, as might be expected from the method of preparation.

My thanks are due to Mr. J. H. Coste for confirming the identification of methyl alcohol.

DISCUSSION.

Mr. F. W. RICHARDSON said he was not sure whether the lowering of the flash-point was entirely due to the methyl alcohol. It seemed remarkable that as little as 0·5 per cent. of such a substance should have the effect of lowering the flash-point to the extent of over 20°, and he should be inclined to think that there must have been some other impurity as well. Personally, however, he did not often determine the flash-point in the case of turpentine.

Mr. A. CHASTON CHAPMAN stated that in his opinion the determination of the flash-point was a very important item in the analytical examination of turpentine.

Mr. NASH said that in a recent paper by Mr. J. H. Coste and himself, the advisability of determining the flash-point of all samples of turpentine was pointed out, and the present case confirmed this, for it was only the flash-point that showed that there was anything abnormal about the sample. Whatever might be the impurity that caused the low flash-point, it was certainly some spirit soluble in water, for when the turpentine was washed the flash-point rose to the normal figure. He should like to add that, when first trying the Conradsen reaction with this Finnish turpentine, he had obtained a negative result, but was so certain that the result ought to be positive that he obtained a fresh supply of sulphurous acid, with which the expected result was obtained. It was therefore evident that the sulphurous acid used must be concentrated. This was mentioned in Conradsen's original paper, but he had never seen the point emphasised.

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NOTE ON THE GRAVIMETRIC ESTIMATION OF PHOSPHORUS IN MILK.

BY E. HOLL MILLER.

(*Read at the Meeting, November* 1, 1911.)

ACCORDING to Söldner, milk contains both primary and secondary phosphates, and also a tertiary phosphate, which is present as tricalcic phosphate. The primary and secondary phosphates are present as mono- and di-potassium phosphate and di-magnesium and calcium phosphate. It would seem highly probable that the temperature at which milk (milk solids) is ignited to obtain the ash, both the primary and secondary phosphates would be converted into the meta and pyro varieties respectively; for when milk is ignited, however careful one is to keep the temperature down during incineration, the temperature actually obtained is considerably higher than that at which primary and secondary phosphates lose water and pass into the meta and pyro states; in fact, it would seem impossible for either a primary or secondary phosphate to exist in a freshly incinerated milk ash. A considerable number of milk ashes have been analysed qualitatively in order to ascertain whether meta- and pyro-phosphates are present, with the final view of determining, if present, their effect on the accuracy of the estimation of phosphorus in milk by any of the ordinary gravimetric methods.

The result of the examination of some four hundred samples of milk ash gave only three which showed the presence of a soluble meta salt; solutions of these three in dilute hydrochloric acid coagulated egg-albumin. A considerable number showed the presence of varying quantities of pyrophosphates; and it was observed that, if during the incineration of the milk the ash was allowed to attain a red heat, pyrophosphates could easily be detected. The presence of pyrophosphate was shown by the solutions giving a bluish-white precipitate with copper sulphate solution, which was insoluble in acetic acid. They also gave a white precipitate with zinc sulphate, soluble in ammonia, and insoluble in acetic acid. These two reactions have been recommended by Arnold and Werner (*Chem. Zeit.*, 1905, **29**, 1326-1327) for the detection of pyrophosphoric acid in the presence of the ortho and meta acids. I may incidentally mention that a sample of milk-powder on incineration gave an ash which contained a very considerable portion of the phosphorus in the meta state. The total quantity of phosphorus in the powder was estimated (by methods mentioned later), and was found to be quite normal. No phosphates of any kind had been used in the preparation of the milk-powder. Very dilute acid solutions of the ash easily coagulated solutions of egg-albumin.

Estimations of phosphorus in a number of milks have been made; the determinations have been carried out on the ash, and the results, when compared with those obtained by treating the ash (in some cases the milk solids) with oxidising agents—*i.e.*, nitric acid, etc.—and then determining the phosphorus in the same manner, have been found to be invariably lower.

The methods used for determinating the phosphorus were the following:

I. By precipitating as magnesium ammonium phosphate after removing the lime as oxalate, and weighing as magnesium pyrophosphate.

II. By precipitating as magnesium ammonium phosphate in the presence of lime, holding the same in solution with ammonium citrate, and weighing as magnesium pyrophosphate.

III. By precipitating as ammonium phosphomolybdate, dissolving and reprecipitating as magnesium ammonium phosphate, and weighing as magnesium pyrophosphate.

IV. By Carius's method.

The following are some of the results obtained:

TABLE I.

	Ash. Per Cent.	CaO. Per Cent.	P_2O_5. Per Cent.
By Method I.			
(*a*) Precipitating as magnesium ammonium phosphate after filtering off lime as oxalate ...	0·72	0·18	0·218
(*b*) Ditto, after heating ash for ninety minutes with strong sulphuric acid	0·72	0·18	0·232
(*c*) Ditto, after heating ash with boiling nitric acid for one hour	0·72	0·18	0·236
(*d*) Ditto, after heating ash with mixture of equal parts of sulphuric and nitric acids for two hours	0·72	—	0·234
By Method II.			
(*e*) Precipitating as magnesium ammonium phosphate in the presence of lime, and keeping the lime in solution with ammonium citrate	0·72	—	0·220
(*f*) Ditto, after heating ash for ninety minutes with strong sulphuric acid	0·72	—	0·230
(*g*) Ditto, after boiling ash with nitric acid for one hour	0·72	—	0·232
(*h*) Ditto, after heating ash with equal parts of sulphuric and nitric acids for two hours ...	0·72	—	0·230

A sample of sweetened condensed milk gave the following results:

	Method I. Per Cent.	Method I. (*c*). Per Cent.	Method II. Per Cent.	Method II. (*g*). Per Cent.
Ash ...	2·004	2·006	2·01	2·004
P_2O_5 ...	0·580	0·670	0·58	0·665

TABLE II.

By Method III.

	Ash. Per Cent.	P_2O_5. Per Cent.
As magnesium pyrophosphate by Method I.	0·71	0·210
By Method II.	0·70	0·207
Ditto, after first heating ash for ninety minutes with strong sulphuric acid	—	0·223
Ditto, after boiling for one hour with strong nitric acid ...	—	0·225
Ditto, after boiling for two hours with mixture of equal parts of sulphuric and nitric acids	—	0·220

A sample of sweetened condensed milk gave the following results by Method III:

	Method III. Per Cent.	Boiling, as above, with Sulphuric Acid. Per Cent.	Nitric Acid. Per Cent.	Sulphuric and Nitric Acids. Per Cent.
Ash ...	1·92	1·920	1·940	1·940
P_2O_5 ...	0·57	0·598	0·621	0·629

An examination of these results shows that there is a small quantity of phosphorus apparently not estimated by Methods I., II., and III., unless the ash is previously treated with some reagent to convert pyro- or meta-, etc., phosphates into the ortho variety. The difference between the results is not very great in the case of milk, but with the samples of sweetened condensed milk the difference is considerably greater. In the case of milk-powder this difference would be greater still. It seems probable that this difference is due to the presence of phosphates in the ash in some condition other than the ortho, which are not precipitated, and thus tend to make the results low. The evidence seems to be conflicting as to whether ordinary magnesia mixture will cause precipitation in solutions of pyro- and meta-phosphates; but even if phosphates in this state are partially precipitated, precipitates of inconstant composition would be obtained which would behave abnormally before the blowpipe, and vitiate results. For these reasons it was thought advisable to compare the results obtained by treating the milk solids by the method of Carius and Method I.:

TABLE III.

Sample.	Ash. Per Cent.	Estimation of P_2O_5 by Method I. in the Ash. P_2O_5. Per Cent.	Treating Milk Solids by Carius's Method, and estimating P_2O_5 as in Method I. P_2O_5. Per Cent.
1	0·75	0·202	0·216
2	0·75	0·218	0·227
3	0·71	0·210	0·223
4	0·74	0·217	0·231
5	0·76	0·211	0·226
6	0·76	0·207	0·222
7	0·75	0·221	0·239

Average difference: 0·014 per cent.

Two samples of sweetened condensed milk gave the following results:

	Ash. Per Cent.	P_2O_5 in Ash. Per Cent.	P_2O_5 by Carius's Method. Per Cent.
1	1·96	0·590	0·63
2	2·04	0·588	0·65

The conclusions drawn from the above results are that the usual methods of estimating phosphorus in milk—viz., Methods I., II., and III.—give results that differ from Carius's method only by about 0·01 per cent. In the case of concentrated and sweetened condensed milk and milk-powders it is evident, however, that it is preferable to treat the milk solids by Carius's method, and estimate the orthophosphoric acid in the solutions so obtained.* It might be mentioned that the close agreement between the results in Table III. seems to show that no appreciable quantity of phosphorus is volatilised during the incineration of the milk. This has also been pointed out by Bordas and Touplain (*Compt. rend.*, 1911, **152**, 899-900).

DISCUSSION.

The CHAIRMAN (Dr. DYER) remarked incidentally that it was not infrequently the case that superphosphate was artificially dried for the purpose of concentrating it, and that in consequence an appreciable proportion of the soluble phosphoric acid was converted into a form that was not orthophosphoric acid, so that low determinations of soluble phosphoric acid might easily be obtained if the precaution of boiling the solution with nitric acid were neglected.

Mr. C. REVIS said that ordinarily he had found very little loss of phosphoric acid to occur in the case of milk, but it was well to bear in mind that the quantity obtained was dependent upon the proportion of lime in the ash. In some experiments which he had recently been making with casein precipitated from milk, he had found the phosphoric acid results to depend upon the quantity of lime that was left in the casein. He could fully confirm the author's remarks as to the Carius method, which he considered to be the most accurate for this purpose. Much the same results were obtained by destroying the organic matter by means of a mixture of hydrochloric acid and nitric acid, but the Carius method was perhaps simpler, and no difficulty was caused by the phosphoric acid being present in different forms.

Dr. MONIER-WILLIAMS considered that Neumann's volumetric method, as modified by Plimmer and Bayliss (*J. of Physiology*, 1906, **33**, 439), was by far the most accurate and expeditious method for the determination of phosphoric acid in any form in food stuffs. He had found the following to be a convenient way of carrying out the determination: A weighed quantity of the substance, which should contain not more than 25 mgrms. of phosphoric acid (P_2O_5) is treated in a round-bottomed Jena glass flask of about 1 litre capacity with 5 c.c. concentrated sulphuric acid, and an excess of strong nitric acid (sp. gr. about 1·40). With some substances the action takes place at once with copious evolution of red fumes, and may be moderated by addition of water. The contents of the flask are strongly heated over a free flame, with constant shaking and repeated addition of small quantities of nitric acid, until a clear liquid is obtained which does not darken on further heating. This operation lasts about ten to fifteen minutes. The liquid is then treated with 70 c.c. of water and 25 c.c. of 50 per cent. ammonium nitrate solution, warmed to about 80° C., and the phosphoric acid precipitated with 25 to 30 c.c. of 10 per cent. ammonium molybdate solution. The mixture is thoroughly shaken and cooled, and the precipitate filtered off by suction through asbestos on a Gooch crucible. It is washed several times with cold distilled water until free from acid, and then transferred together with the asbestos to an Erlenmeyer flask, with a small quantity of distilled water. $\frac{N}{2}$ NaOH is added from a burette in quantity more than sufficient to dissolve the precipitate, the liquid is boiled until all the ammonia has been driven off, cooled, and titrated back with $\frac{N}{2}$ H_2SO_4 and phenolphthaleïn. One c.c. $\frac{N}{2}$ NaOH $= 0{\cdot}001268$ grm. P_2O_5. He had used this method for a great many determinations of phosphoric acid in flour, and had found it to be quicker and more accurate than the gravimetric methods, while there could be no formation of meta- or pyro-phosphate. It was also, in his opinion, much better than the Carius method, and, as far as he could see, could be used for the determination of phosphoric acid in any form, organic or inorganic.

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* After washing out the contents of the Carius tube, it is advisable to take the liquid down to dryness on the water-bath in order to get rid of excess of HNO_3. The residue can then be taken up in water, with the addition of a small quantity of HCl if necessary.

Mr. Bevan said that the working of the Neumann method had been shown to him by Dr. Monier-Williams, whose observations with regard to it he could fully confirm. He did not, however, quite understand why it should be necessary to eliminate the sulphuric acid by the addition of ammonium nitrate, because in some molybdic solutions sulphuric acid was deliberately used.

Dr. Monier-Williams thought it probable that the composition of the precipitate was constant only in presence of a large excess of nitric acid, and that if nitric acid were not present the ratio of ammonia to phosphoric acid might be different. Of course, where the precipitate was redissolved and weighed as magnesium phosphate, this ratio was of no moment.

Mr. W. Bacon said that in those processes in which sulphuric acid was allowed to be present, the solution was not heated beyond 40° C. At the higher temperature in Neumann's method the sulphuric acid might cause the precipitation of molybdic acid, which was perhaps the reason for insisting on its removal.

Mr. Richmond said that it seemed to him that in normal milk (*i.e.*, milk which had not become sour), although monobasic and dibasic phosphates were present, there was practically always sufficient base in combination with organic acids, to form orthophosphates with all the phosphoric acid on ignition. If, however, the milk contained even sufficient acid to cause it to curdle on heating, there was a disturbance of the conditions. When casein was precipitated by means of acid, the quantity of base remaining was insufficient to form orthophosphates with the whole of the phosphorus, organic and inorganic, so that, when separation of the casein took place (which of course would be local), there would be an excess of phosphoric acid and a deficiency of base, and meta- and pyro-phosphates would be formed. The proportions of soluble and insoluble ash in milk before and after souring were distinctly different.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Detection of Benzoic Acid in Foods, Etc. E. Polenske. (*Arbeit. Kaiserl. Gesundheitsamte*, 1911, **38**, 149-154; through *Chem. Zentralbl.*, 1911, II., 1179-1180.)

I. *Estimation of the Total Benzoic Acid Occurring in Cranberries.*—The method proposed consists of the following operations: (1) Extraction of the cranberries with 96 per cent. alcohol for one hour at a temperature of 70° C.; (2) separation of the impure benzoic acid; (3) purification of the same by treatment with potassium permanganate as described by von der Heide and Jakob (ANALYST, 1910, **35**, 166); and (4) sublimation and titration of the pure benzoic acid. The alcoholic extract of the fruit is rendered strongly alkaline with sodium hydroxide, filtered, the residue washed with alcohol, and the filtrate evaporated until all alcohol has been removed. The residual solution is acidified with sulphuric acid, and extracted with a mixture of ether and petroleum spirit, the benzoic acid being then obtained in aqueous solution by shaking the ethereal extract with sodium hydroxide solution. This alkaline benzoate solution is now heated and oxidised with potassium permanganate as described (*loc. cit.*), and, after the excess of permanganate employed has been decomposed by the addition of sodium sulphite and sulphuric acid, the benzoic acid is extracted from the solution with ether. The ethereal solution is filtered, the ether is evaporated, the residue is dissolved in a small quantity of ether, and the solution is transferred to a test-tube. After evaporating the ether, the dry residue is mixed with a quantity of sand, and heated in an oil-bath at 180° to 190° C. until the benzoic acid has sublimed on to the upper part of the tube. The bottom of the tube is then cut off, the crystals of benzoic acid are dissolved in alcohol, and the solution is titrated, using phenolphthaleïn as indicator; 1 c.c. of $\frac{N}{10}$ alkali solution is equivalent to 0·0122 grm. of benzoic acid. The total quantity of benzoic acid occurring naturally in cranberries was found to vary from 0·089 to 0·206 per cent., according to the degree of ripeness of the fruit.

II. *Detection of Benzoic Acid in Wine.*—After the wine has been evaporated and acidified, as described by von der Heide and Jakob (*loc. cit.*), it should be filtered before the extraction with ether in order to prevent the formation of a troublesome emulsion. The separated benzoic acid must be treated with permanganate to destroy any salicylic acid, etc., which may be present, and the benzoic acid is then identified by converting it into salicylic acid by fusion with potassium hydroxide and applying the ferric chloride test.

W. P. S.

Estimation of Benzoic Acid. O. Folin and F. F. Flanders. (*J. Amer. Chem. Soc.*, 1911, **33**, 1622-1626.)—The authors have adapted the modified method of La Wall and Bradshaw, used by them in the estimation of benzoic acid in cranberries, to the case of ketchup, using the chloroform titration method proposed by Folin and Wentworth (*J. Biol. Chem.*, 1908, **7**, 423) as the final stage. Twenty-five grms.

of ketchup are well mixed with 2 c.c. of strong nitric acid, and several small portions of sodium nitrite (about 0·2 to 0·3 grm. in all) added. These additions serve to destroy colouring matters, and to prevent subsequent emulsification. The bleached mass is transferred, without previous filtration, to a separator by means of 200 c.c. of a saturated solution of ammonium sulphate, and then extracted with successive quantities of 50, 35, 25, 25, and 25 c.c. of chloroform, the shaking being done by means of a gentle rotary motion. The mixed chloroform extracts are shaken twice with 200 c.c. each time of a saturated solution of sodium chloride containing 3 c.c. of hydrochloric acid (10 per cent.) per litre. The washed chloroform is then titrated with standard sodium alcoholate solution, using 4 or 5 drops of alcoholic phenolphthaleïn as indicator, to the first persistent pink colour. The standard alcoholate is prepared by dissolving 2·3 grms. of cleaned metallic sodium in 1 litre of absolute alcohol, and is standardised against pure benzoic acid in 160 c.c. of chloroform. Any hydrochloric acid in the chloroform used for standardisation should be got rid of just before use. Washing of the chloroform extract by salt solution removes glycolic, lactic, and acetic acids, but cinnamic acid, if present, will be estimated with the benzoic acid. The method may be adapted for the qualitative detection of benzoic acid. A. R. T.

The Dimethyl Sulphate Test for Creosote Oil and Creosote Baths (Dips). R. M. Chapin. (*U.S. Dept. Agric. Circular*, No. 167; through *Chem. Zentralbl.*, 1911, II., 1386-1387.)—The purpose of the test is to detect the presence of paraffin hydrocarbons in tar oils. The oil to be tested is submitted to fractional distillation, and 5 c.c. of a high-boiling fraction are shaken in a narrow tube with 8 c.c. of dimethyl sulphate; the lower layer will then consist of dimethyl sulphate containing the aromatic compounds, whilst the paraffins, if present, form the upper layer (*cf.* Valenta, ANALYST, 1906, **31**, 202). This test may be used in place of the sulphonation test, or, in important cases, employed in conjunction with the latter. In testing creosote baths (dips or washes), 50 grms. of the sample are treated with potassium hydroxide and extracted with ether; the ethereal extract is evaporated, dried, and tested with dimethyl sulphate. Should the result be negative, the sample contains neither creosote of doubtful origin, nor petroleum oils. If a positive result be obtained, a portion of the sample should be distilled, and the fraction boiling at 240° to 270° C., tested with dimethyl sulphate. (*Cf.* Harrison and Perkin, ANALYST, 1908, **33**, 2.) W. P. S.

Estimation of Gelatin. W. Greifenhagen, J. König, and A. Scholl. (*Biochem. Zeitsch.*, 1911, **35**, 217-227.)—As the results of an examination of various methods which have been proposed for the estimation of gelatin, the authors find that the precipitation method with formaldehyde described by Beckmann (ANALYST, 1895, **20**, 44) is useless. Precipitation with Nessler's reagent in the presence of a tartrate yields quantitative results, but when proteoses (albumoses) are present, these are also precipitated. Trichloracetic acid in large excess yields a turbidity with very dilute solutions of gelatin, and does not precipitate proteoses completely. Mercuric chloride precipitates proteoses from neutral solutions, but not gelatin; the proteoses are not, however, precipitated completely. If from a solution the gelatin and proteoses be precipitated together by means of zinc sulphate, the precipitate then dissolved, and treated with mercuric chloride, the nitrogen-content of the mercuric chloride precipitate affords evidence of the presence of gelatin; should it contain approximately the same amount of nitrogen as the zinc sulphate precipitate, gelatin is not present, but if there be a considerable difference between the two quantities of nitrogen, gelatin is probably present. In the latter case, a second portion of the solution under examination is treated with zinc sulphate, the precipitate formed is redissolved, and treated with a solution of mercuric iodide in either alcohol or acetone; if this precipitate contains much nitrogen, as compared with that present in the zinc sulphate precipitate, the presence of gelatin is confirmed. These methods, however, give only approximately quantitative results. W. P. S.

Detection of Invert Sugar in Honey. E. Feder. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 412-413.)—The test proposed depends upon the detection of α-hydroxyl-δ-methyl-furfural by means of aniline chloride, and is applied directly to the honey and not to the ethereal extract of the same as in Fiehe's test (ANALYST, 1908, **33**, 397; 1909, **34**, 399). Five grms. of the honey are mixed thoroughly in a porcelain basin with 2·5 c.c. of freshly prepared aniline chloride solution (100 c.c. of aniline and 30 c.c. of 25 per cent. hydrochloric acid). Should invert sugar be present, a bright red coloration develops almost immediately, its intensity being dependent on the quantity of α-hydroxyl-δ-methyl-furfural contained in the invert sugar. Pure honey yields a yellowish coloration with the test. W. P. S.

Estimation of Water in Honey. U. Fabris. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 353-358.)—The author has investigated various methods which have been proposed for the estimation of water in honey—namely: (1) Drying on sand *in vacuo* at 100° C.; (2) drying on sand *in vacuo* at 60° C.; (3) distillation with turpentine and measuring the volume of water distilling over with the turpentine; (4) drying at 95° C. on strips of paper for two and a half hours, the strips of paper being contained in a specially constructed tube through which a current of dry air is passed during the operation. The results obtained by the four methods are shown in the following table:

	Five Hours *in vacuo* at 100° C.	Twenty-eight Hours *in vacuo* at 60° C.	Distillation Method.	Drying in a Current of Air.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Sample 1	13·05	12·60	12·68	12·65
„ 2	18·72	18·25	18·30	18·32
„ 3	23·75	23·43	23·52	23·47

The first method always yields higher results than the others, whilst in the case of the distillation method it is somewhat difficult to obtain concordant results in duplicate estimations. W. P. S.

Valuation of Hops by Analysis. E. Moufang and A. Scheer. (*Wochsch. Brau.*, 1911, **28**, 449-452; through *J. Soc. Chem. Ind.*, 1911, **30**, 1226.)—The authors give the results of investigations on the determination of the extract of hops. A special straining vessel for holding the finely ground hops during mashing was used. This vessel, holding 20 to 25 grms. of material, is made of perforated sheet nickel, and is fitted with a vertical rod, the lower end of which is fixed to the bottom of the vessel. A perforated cover fits the interior of the vessel, and is provided with a collar which slides on the rod. The cover thus serves to press the hops after extraction, and the vessel serves to stir the mashing water. It is found that the best results (*i.e.*, the maximum yields of extract) are obtained by mashing 20 grms. of the finely divided hops with 265 c.c. of water at a temperature of 90° C. for fifty-five to sixty minutes; to allow for loss by evaporation, about 40 c.c. of water should be added during mashing. The amount of water required to make up the weight of the cooled mash must not exceed 3 to 5 grms., and instead of this, it seems advantageous to add only enough water to complete the weight to the next gram. Prior to analysis, the hops should be freed from stalks and foreign impurities, and thoroughly divided. The limit of error in the determination is about 0·2 to 0·3 per cent. Analyses of a number of hops from various sources showed that the extract contents ranged from 28·76 to 36·25 per cent., and corresponded closely with the price of the hops. In conjunction with the determination of extract, the aroma and colour of the hops may also be judged by the above method.

Estimation of Morphine by Extraction with Phenyl-ethyl Carbinol. A. D. Thorburn. (*J. Ind. Eng. Chem.*, 1911, **3**, 754-756.)—A quantity of the sample representing less than 0·175 grm. of anhydrous alkaloid is dissolved in a few c.c. of acidulated water; three-sevenths of its volume of ethyl alcohol are added to the solution, which is then filtered and transferred to a separating funnel with sufficient 30 per cent. alcohol to make a total volume of 10 to 15 c.c. The liquid is made alkaline with ammonia and a mixture of 3 c.c. of phenyl-ethyl carbinol with 1 c.c. of benzene is added. The contents of the funnel are shaken hard for several minutes, allowed to separate, and again shaken for several minutes, after which the two liquids are separated. The aqueous liquid is returned and extracted again with half the above quantity of mixed solvents. The alkaloidal extract should be heated in a bath of boiling water for an hour, and the residue poured into a separating funnel, washing with ether, a little at a time, until 30 c.c. have been added. A known volume (10 c.c.) of $\frac{N}{10}$ sulphuric acid is added, the liquid being shaken at intervals during half an hour. The aqueous layer is drawn off, the liquid in the funnel being washed with 3 c.c. of water three times. The morphine is finally estimated by titrating back the excess of sulphuric acid in presence of hæmatoxylin. J. F. B.

Estimation of Nitrates in Milk by Means of the Diphenylamine-Sulphuric Acid Test. J. Tillmans and A. Splittgerber. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 401-405.)—Certain modifications are recommended for the purpose of rendering more trustworthy the method described previously by one of the authors (ANALYST, 1911, 67). The protein and fat-free serum of the milk is best prepared by treating the milk with mercuric chloride and hydrochloric as described in the process for estimating sulphates in milk (ANALYST, 1910, **35**, 434); 1 c.c. of the serum thus obtained is mixed with 4 c.c. of the diphenylamine reagent and any blue coloration which develops is compared with that produced by solutions containing known amounts of nitrates. As the milk serum still contains substances which diminish the intensity of the coloration produced by the reaction, these standard solutions should be prepared by placing quantities of 0·45, 0·8, 1·2, 1·5 and 2·0 c.c. of potassium nitrate solution, containing 0·1871 grm. of the salt per litre, in separate 100 c.c. flasks, adding 2 c.c. of saturated sodium chloride solution to each, and diluting the mixtures with water to the mark. The solutions then contain quantities of nitric acid (N_2O_5) corresponding with 1, 2, 3, 4, and 5 mgrms. of N_2O_5 per litre of milk, respectively. W. P. S.

Estimation of Starch. W. Greifenhagen, J. König, and A. Scholl. (*Biochem. Zeitsch.*, 1911, **35**, 194-216.)—The polarimetric methods for the estimation of starch proposed by Lintner (ANALYST, 1907, **32**, 179) and Ewers (ANALYST, 1908, **33**, 101, 481) may be used for all kinds of starch; in Lintner's method the different starches have practically the same specific rotation, namely, +202°. This value varies slightly for different starches in Ewer's method, but for maize, rice, wheat, rye, barley, and oats, an average value of $[\alpha]_D$ +183·4° may be taken. Feeding stuffs and other products containing starch should be washed with cold water, alcohol, and ether before the estimation is commenced, in order to remove optically active substances other than starch; cellulose, hemi-cellulose, and pentosans are without influence on the results obtained by either method. Ewer's method is the more applicable to the estimation of starch in pepper, whilst Lintner's method may be used in the case of cocoa and cinnamon; the latter substance should be washed with hot alcohol previous to the estimation of the starch. W. P. S.

Estimation of Sucrose and the Detection of Dextrin in Foods. F. Vollant. (*Ann. Falsific.*, 1911, **4**, 504-509.)—The author has made a number of estimations of sucrose in such substances as commercial sugars, syrups, confectionery, sweetened milks, honey, etc., using the process described by Lemeland (ANALYST, 1910, **35**, 539), the results obtained showing that the process is trustworthy. After the sugar solution has been treated with manganese dioxide and alkaline hydrogen peroxide solution as prescribed, it is cooled and neutralised by the addition of acetic acid (*not* nitric acid as stated in the original paper), basic lead acetate is added, the mixture is diluted to a definite volume (100 c.c.), and filtered. Fifty c.c. of the filtrate are acidified with acetic acid, diluted to a volume of 55 c.c., and examined by the polarimeter. A sample of commercial sugar was found to contain 91·14 per cent. of sucrose, whilst the ordinary inversion method yielded 91·28 per cent. In the case of sweetened dry milk, 5 grms. of the sample are extracted with three successive quantities of 25 c.c. of 0·1 per cent. acetic acid, the insoluble matter being separated each time by centrifugal action; the united extracts are treated with 3 c.c. of a 25 per cent. sodium metaphosphate solution, and 3 c.c. of hydrochloric acid, diluted to a volume of 100 c.c., and filtered. An aliquot portion of the filtrate is then acidified with acetic acid, diluted to a definite volume, and polarised. A sample of this product yielded 14·0 per cent. of sucrose against 13·92 per cent. found by the inversion method. For the detection of dextrin in food materials, proteins and gums are removed by the usual methods, the sucrose which may be present is inverted, and the resulting solution is treated as described; any optical property it now exhibits is due solely to the presence of dextrin. W. P. S.

Essential Oil of Santolina Chamæcyparissus. L. Francesconi and Scarafia. (*Gazz. Chim. Ital.*, 1911, **41**, 185-203.)—The medicinal plant, *Santolina chamæcyparissus*, which is found in dry places in the Mediterranean provinces of Italy, contains a yellow essential oil, the yield of which was found to vary from 0·19 to 1·15 per cent., according to the season of distillation. Oil distilled in June had the following characteristics: Sp. gr. at 15° C., 0·8704; $[\alpha]_D = -11·66°$; saponification value, 11·78; and saponification value of acetylated oil, 11·74. On fractional distillation it yielded two main fractions at 94° to 97° C., and 98° to 101° C., respectively, a third smaller fraction at 102° to 113° C.; and a smaller fraction at 114° to 135° C. The oil contained neither free alcohols nor phenols, and only a small quantity of compound esters. It contained a terpene which was abundant in the first fraction, together with a phenolic ester; in the middle fractions there were one or more oxyterpenes and carbonylic compounds; whilst the last fraction contained other carbonylic compounds and the ester of an alicyclic alcohol. C. A. M.

Spanish Oil of Turpentine. O. Fernandez. (*Chem. Zeit.*, 1911, **34**, 1152.)—The principal constituent of Andalusian pine oil (*Pinus Halepensis*) is a terpene which is not quite identical with ordinary pinene. From Spanish pinene no nitrosite could be obtained. By the action of nitrogen peroxide at 0° C. in presence of acetic acid, a brownish-black substance was separated, which had an odour resembling that of cymene, amounting to about 55 per cent. of the original hydrocarbon. This is probably a form of oxime, becoming crystalline in course of time. The corresponding pinenenitrolpiperide forms a spongy, non-crystalline mass. With toluidine a viscous liquid boiling at 130° to 141° C. under 14 mm. pressure is obtained; α- and β-naphthylamine and sulphanilic acid yield no nitrolamines. Only about 10 per cent. of a crystalline terpene could be obtained from the Spanish pinene.

J. F. B.

Cider Vinegar. F. E. Mott. (*J. Ind. and Eng. Chem.*, 1911, **3**, 747-750.)—The author summarises the results of his investigations as under: Assuming that the sugars naturally present in pure cider vinegar are essentially lævulose and dextrose, a relation exists between the percentages of these two sugars in a pure cider vinegar. If the reducing sugars be determined with elimination of the error due to aldehyde, it is believed that in all pure cider vinegars the percentage of dextrose in the total solids is less than 0·6 times the percentage of lævulose. Whenever a cider vinegar is dextro-rotatory at 87° C., sucrose being absent, it may be inferred that it has been adulterated with foreign dextro-rotatory materials, usually dextrose; but a vinegar may be adulterated with such material and yet show a lævo-rotation at 87° C. Whenever a value for dextrose per cent. of solids is found in excess of 0·6 times the lævulose value, the sample is not exclusively the fermented product of pure apple-juice, but, sucrose being absent, has been adulterated with some foreign dextro-rotatory material. The method ultimately adopted for the determination of the analytical data is as follows:

Total Solids.—Ten c.c. of the vinegar are evaporated to dryness on the boiling water bath in a flat-bottomed platinum dish about 60 mm. in diameter. Five c.c. of water are added and again evaporated to dryness; again 5 c.c. of water are added and evaporated, and the dish is allowed to remain for one hour on the water-bath after the final evaporation. The dish is removed, wiped, and placed in an oven at 100° C. for five minutes, cooled in the desiccator, and weighed.

Reducing Sugars.—Twenty-five c.c. of water are added to the dry solids in the dish and heated till the latter are dissolved. The contents of the dish are rinsed into a 100 c.c. flask, cooled, neutralised, and made up to the mark. The reducing sugars are then determined by Munson and Walker's method on 50 c.c. of the solution, and calculated as dextrose.

Polarisation.—To 50 c.c. of the vinegar 5 c.c. of basic lead acetate, sp. gr. 1·25, are added, and two teaspoonfuls of animal charcoal which has previously been tested as to its suitability for polarisation purposes. The liquid is filtered and polarised in a 200 mm. tube, the result being expressed in Ventzke degrees corrected. The percentage of lævulose is then calculated by the formula $L = \frac{0{\cdot}793R - P}{2{\cdot}08}$, where R = per cent. of reducing sugars, as dextrose, in the total solids, and $P = \frac{p \times 26{\cdot}048}{\text{total solids}}$, where p is the polarisation observed as above.

The percentage of dextrose, $D = R - 0{\cdot}915L$. J. F. B.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Comparative Values of the Biological Reactions with Serum-Precipitin and the So-called Erythro-Precipitin for the Detection of Blood. D. Mirto. (*Arch. Farmacol. sperim.*, 1911, **12**, 145-158; through *Chem. Zentralbl.*, 1911, II., 1272.)—The results of an investigation regarding the sensitiveness and specific properties of these two reactions are recorded. The serum-precipitin method yields a positive reaction with the serum and hæmoglobin portion of blood, whilst the erythro-precipitin test (preparation of a precipitin-serum for human blood by the intraperitoneal injection of human blood-corpuscles into rabbits) only reacts with the hæmoglobin of the blood and not with the proteins of serum, semen, milk, or saliva; it is less sensitive than the serum-precipitin test, and does not give a reaction in the absence of oxyhæmoglobin. The serum-precipitin test is considered to be the more reliable test. W. P. S.

Use of Elastin in the Detection of Proteolytic Enzymes. E. Abderhalden and K. Kiesewetter. (*Zeitsch. physiol. Chem.*, 1911, **74**, 411-426.)—The use of elastin for the detection of active pepsin in the contents of the intestines has been described previously (ANALYST, 1911, 551) by Abderhalden and Meyer, and the authors now give the results of further experiments, showing the suitability, etc., of elastin for the purpose in question. They have also examined human, dogs' and horses' fæces as to the presence of proteolytic ferments; whilst small quantities of the latter sometimes appeared to be present, in many cases negative results were obtained. Further experiments proved the presence of peptolytic ferments in the plasma obtained from horses' livers and kidneys, and from yeast. W. P. S.

Occurrence, Detection, and Estimation of Nitrous Oxide in the Gases produced by Denitrifying Organisms. S. Suzuki. (*Zentralbl. Bakter. Parasitenk Zweite Abth.*, 1911, **31**, 27-49; through *Chem. Zentralbl.*, 1911, II., 1267-1268.) —It is known that bacteria are capable of producing nitrous oxide by the reduction of nitrates, and the author gives the following method of identifying this gas in the presence of the other gases produced at the same time : The gaseous mixture is collected in an ordinary explosion pipette over dry mercury, diluted with its own volume of air, and subjected to the action of a small electric spark. If the gases be then passed over potassium iodide-starch solution, a blue coloration will be obtained. The presence of 1 per cent. of nitrous oxide in the gases may thus be detected. The quantity of the nitrous oxide may be estimated by diluting the gases with pure nitrogen (if less than 10 per cent. of nitrous oxide be present this dilution is unnecessary), the carbon dioxide is removed by treatment with potassium hydroxide, the oxygen with pyrogallol, and the gaseous residue is heated in a platinum capillary tube after being mixed with carbon monoxide free from carbon dioxide and oxygen. The volume of the resulting carbon dioxide corresponds with the volume of the nitrous oxide. W. P. S.

Colorimetric Estimation of Sugar, Creatine, and Creatinine in Urine. W. Autenrieth and G. Müller. (*Münch. med. Wochenschr.*, 1911, **58**, No. 17; through *Chem. Zentralbl.*, 1911, II., 1380-1381.)—Whilst the process described by Autenrieth and Tesdorpf (ANALYST, 1910, **35**, 528) yields accurate results when applied to colourless urine, certain modifications are necessary in the case of dark-coloured urine. The use of blood-charcoal free from iron is recommended in place of lead acetate for removing the colouring matters, and this treatment is carried out after the diluted urine has been boiled with Bang's copper solution (ANALYST, 1907, **32**, 180); the blood-charcoal when free from iron does not retain any of the copper. Fifty c.c. of Bang's solution and 10 c.c. of the diluted urine (1 volume of urine to 4 volumes of water, or 10 volumes of water if much sugar be present) are boiled for three minutes, cooled, and diluted with potassium thiocyanate to a definite volume. To the mixture is now added 0·5 grm. of blood-charcoal, and, after shaking for ten minutes, the mixture is filtered, the amount of unreduced copper being then estimated by comparing the filtrate in a colorimeter with standard copper solutions, or other standards. The creatinine and creatine may be estimated by means of Folin's colorimetric process. W. P. S.

Colorimetric Estimation of Lactose in Urine and in Milk. W. Autenrieth and A. Funk. (*Münch. med. Wochenschr.*, 1911, **58**, No. 17; through *Chem. Zentralbl.*, 1911, II., 1382.)—In the case of urine, the actual presence of lactose should be ascertained by heating 5 c.c. of the urine with 4 c.c. of 10 per cent. ammonia and 4 drops of potassium hydroxide solution; should lactose be present, a red coloration is produced. Dextrose yields a brown coloration with the test when present in larger quantity than 1 per cent.; smaller quantities do not yield a coloration, nor do they interfere with the lactose reaction. For the estimation of lactose 10 c.c. of urine (containing not more than 0·04 grm. of lactose) are boiled with 50 c.c. of Bang's copper solution (ANALYST, 1907, **32**, 180), cooled, diluted with potassium thiocyanate solution to a definite volume, 0·5 grm. of blood-charcoal free from iron is added, and the mixture is filtered. The quantity of unreduced copper in the filtrate is then estimated by comparison with standard solutions in a colorimeter. Lactose may be estimated in milk by diluting 25 c.c. of the sample with about 400 c.c. of water, adding acetic acid, boiling the mixture, and diluting it, when cold, to a volume of 500 c.c. After filtration 10 c.c. of the filtrate are boiled with 50 c.c. of Bang's solution, the estimation being then carried out as described above. W. P. S.

Method of Destroying Organic Matter by Means of Bromine, particularly applicable to Toxicological Analysis. G. Magnin. (*J. Pharm. Chim.*, 1911, **4**, 302-306.)—It is recommended that the substance under examination, such as portions of animal organs, etc., be heated together with bromine on a water-bath. The organic matter is thus destroyed rapidly, and a clear solution obtained, which may then be tested for the presence of metallic poisons. W. P. S.

ORGANIC ANALYSIS.

Colorimetric Test for Alcohol in the Presence of Acetone. H. Agulhon. (*Bull. Soc. Chim.*, 1911, [iv.], **9**, 881-885.)—A solution of 0·5 grm. of potassium bichromate in 100 c.c. of pure nitric acid gives an immediate violet-blue coloration in the cold with organic compounds containing certain readily oxidisable groupings, whilst on heating there is a green coloration which changes to blue on cooling. These reactions, which are due to the formation of a chromium nitrate, are given in the cold by aldehydes, alcohols, and compounds containing an alcoholic group, such as sugars, hydroxy fatty acids, etc. Formaldehyde and alcohol react within one or two minutes when treated with 3 c.c. of the reagent to 1 c.c. of a solution of 0·5 per cent. strength and upwards. The test may be used for a quantitative colorimetric estimation of alcohol in the presence of acetone. An analogous but more restricted reaction is obtained by the use of a hot solution of 0·5 grm. of potassium bichromate and 50 grms. of potassium bisulphate in 50 c.c. of water. Green colorations are produced by alcohol, ether, and bodies containing alcoholic or aldehydic groups, but not by unsaturated fatty acids, such as oleic and linoleic acids. On the other hand, ricinoleic acid gives the reaction, and the test may be used for the detection of castor oil in the presence of other fatty oils. It may also be used for the detection of the ketones of cyclohexane. C. A. M.

Differentiation between Amino-Acids and Peptones or Peptides. P. A. Kober. (*J. Biol. Chem.*, 1911, **10**, 9-14; through *Chem. Zentralbl.*, 1911, II., 1063-1064.)—The fact that the copper salts of amino-acids separate copper hydroxide on boiling with alkali, whilst the copper compounds of peptones and peptides are not decomposed on such treatment, may be made to serve for the detection of amino-acids among the products of protein hydrolysis, and for the study of the quantitative relationships which exist between the nitrogenous substances of greater or less complexity at successive stages in an experiment with proteolytic enzymes. The neutral or very faintly alkaline solution is boiled for fifteen minutes with an excess of copper carbonate or of freshly precipitated copper hydroxide, and then filtered. If cuprous oxide separates during the boiling with copper hydroxide, the solution is filtered, neutralised, and reboiled with more copper hydroxide, and again filtered. The filtrate is rendered distinctly alkaline by addition of 5 to 10 c.c. of $\frac{N}{10}$ alkali, and again boiled when, if amino-acids are present, copper hydroxide separates.

G. C. J.

Estimation of Camphor. H. C. Fuller. (*J. Ind. and Eng. Chem.*, 1911, **3**, 791.)—The following adaptation of the hydroxylamine method for the estimation of ketones is described as applied to the determination of camphor in spirits of camphor: Twenty-five c.c. of the sample are measured out into an Erlenmeyer flask, 2 grms. of sodium bicarbonate are added, and then 35 c.c. of a solution of hydroxylamine (20 grms. of the hydrochloride, 30 c.c. of water, and 125 c.c. of absolute alcohol free from aldehyde). The flask is connected with a reflux condenser, and the liquid heated to boiling for two hours. It is then cooled, treated with a mixture of 6 c.c. of hydrochloric acid (1·12 sp. gr.) and 6 c.c. of water, transferred to a 500 c.c. graduated flask, and diluted to the mark; 50 c.c. are measured out, neutralised in presence of methyl orange, phenolphthaleïn is then added, and the excess of hydroxylamine is titrated with $\frac{N}{10}$ alkali. A blank experiment is made, using the same quantity of hydroxylamine solution and 25 c.c. of alcohol to correspond with the spirits of camphor, and the difference between the two titrations represents the hydroxylamine combined with the camphor; 1 c.c. of $\frac{N}{10}$ sodium hydroxide is equivalent to 0·01509 grm. of camphor. J. F. B.

Estimation of Carbon and Nitrogen in Organic Compounds. E. P. Frankland. (*J. Chem. Soc.*, 1911, **99**, 1783-1785.)—The Frankland-Armstrong vacuum-combustion process can be adapted to the estimation of carbon and nitrogen in organic compounds, the carbon dioxide and nitrogen produced in the combustion being measured together, and the latter found by absorption of the carbon dioxide with caustic alkali. The presence of halogens does not affect the results, and only very small quantities of substance are necessary (0·1 grm., or less).

Besides the Sprengel pump, two glass measuring-tubes of 150 c.c. and 50 c.c. capacity, graduated in $\frac{1}{10}$ c.c., are required, and glass cylinders in which the tubes may be levelled over mercury and water respectively. The substance is first mixed with fine copper oxide, and placed in a combustion-tube closed at one end, and the tube then nearly filled with coarse copper oxide. A reduced copper spiral is fitted in, and the tube then drawn out by the blowpipe, attached to the pump and exhausted, and towards the end of the exhaustion some burners below the copper spiral are lighted. The usual method is followed, and the gases evolved collected over mercury in a tube with a tap at one end. When the evolution of gas has ceased, the combustion-tube is exhausted, and the measuring-tube transferred to a cylinder of mercury, where the volume of moist gas (carbon dioxide and nitrogen) is measured. The results are accurate. A. R. T.

Determination of Acids in Sugar-Cane Juice. P. A. Yoder. (*J. Ind. Eng. Chem.*, 1911, 3, 640-647.)—A critical review of the methods available for the separate estimation of citric, aconitic, succinic, malic, tartaric, and oxalic acids in presence of each other and of large quantities of sugar and cane-juice. An apparatus for the continuous extraction of an aqueous solution by an immiscible solvent of greater or less density is figured, and its application to the ethereal extraction of succinic and aconitic acids described. It is shown that aconitic acid shares with citric acid the property of forming a calcium salt less soluble in hot than in cold water, and, further, that the properties of barium aconitate are such that, when Jörgensen's method for the differentiation of organic plant acids (*Zeitsch. Unters. Nahr. Genussm.*, 1907, 13, 241) is applied, aconitic acid is estimated partly as citric and partly as malic acid. Experiments are described which make it doubtful whether tartaric, succinic, or citric acid are present in cane-juice: Aconitic acid is certainly the preponderant acid, and malic and oxalic acids are certainly present, though in very small amount, their sum bearing the ratio of about 1 : 100 to the amount of aconitic acid. The absence of citric acid is by no means certain, owing to the difficulty of differentiating between it and aconitic acid, and the author's work did not take account of tricarballylic acid, found by von Lipmann in sugar beets, nor of lactic acid.

G. C. J.

Detection of Small Quantities of Disaccharides. C. Neuberg and S. Saneyoshi. (*Biochem. Zeitsch.*, 1911, 35, 44-55.)—The process described depends on the different behaviour of the osazones of maltose, isomaltose, lactose, and melibiose, towards hydrolytic agents, and may be applied to as small a quantity as 0·01 grm. of the osazones. The osazones are first prepared in the usual way ; the presence of maltosazone is detected by treating a solution or emulsion of the osazones with yeast-maltase; dextrose is formed, should the mixture contain this osazone, and the resulting solution reduces Fehling's solution. Isomaltosazone is not attacked by yeast-maltase, but by boiling with 1·5 per cent. sulphuric acid it is converted into dextrose, which may be identified from its rotation, reducing power, etc. Whilst lactosazone and melibiosazone are not acted on by yeasts, they are converted into *d*-galactose when heated with 1·5 per cent. sulphuric acid, and the *d*-galactose may be identified by its characteristic behaviour towards yeasts or enzymes, and by its rotation. It is not possible to distinguish between these two osazones, but it is unlikely that they will be found together in any liquid under examination, as lactose is obtained from the milk of animals, whilst melibiose is of vegetable origin.

W. P. S.

Estimation of Free Fatty Acids in the Presence of Soaps of the Alkalis and Alkaline Earths. D. Holde and J. Marcusson. (*Zeitsch. angew. Chem.*, 1911, **24**, 1945-1948.)—Of recent years fats containing considerable amounts of soaps of the alkalis and alkaline earths have been sold under the name of "Calypso fats," and the presence of these substances has an influence upon the quantities of solvent required to dissolve the free fatty acids. Experiments, which are described in detail, have shown that the acid values of such fats may be accurately estimated as follows: In the case of ordinary greases, 10 grms. of the sample are heated beneath a reflux condenser with 50 c.c. of a mixture of 9 parts by volume of petroleum spirit and 1 part of absolute alcohol, the solution filtered while hot, and the filtrate and washings shaken in a separating funnel with 30 c.c. of 50 per cent. (by volume) alcohol, and titrated with standard alkali until the lower layer appears red to phenolphthaleïn. For "Calypso fats" 100 c.c. of a mixture of petroleum spirit and alcohol (8 : 2) are used, a further addition of 10 c.c. of alcohol being made if required to dissolve the fat, and the solution is treated as described above. C. A. M.

Estimation of Formaldehyde. E. Elvove. (*Amer. J. Pharm.*, 1911, **83**, 455-471.)—The potassium cyanide method of estimating formaldehyde is commonly accepted as being only applicable to very dilute solutions. It is here shown, however, that it is more reliable than the hydrogen peroxide method for concentrated solutions, and that it allows of the estimation of formaldehyde in the presence of other aldehydes. Variations in the temperature do not affect the results provided that an excess of potassium cyanide of as much as half an equivalent is added. In using the method for the analysis of the ordinary 40 per cent. formalin solution, 0·5 grm. of the sample is weighed into a stoppered Erlenmeyer flask of about 150 c.c. capacity, and immediately treated with 100 c.c. of a standardised solution of potassium cyanide (about 6·5 grms. per litre). After thorough mixing, the contents of the flask are added to a mixture of 40 c.c. of $\frac{N}{10}$ silver nitrate solution and 10 c.c. of dilute (10 per cent.) nitric acid, the washings added, and the whole made up to 200 c.c., thoroughly shaken, and filtered. The excess of silver in 100 c.c. of the filtrate is then titrated with $\frac{N}{10}$ ammonium or potassium thiocyanate, with ferrous ammonium sulphate as indicator. The amount of potassium cyanide, in terms of $\frac{N}{10}$ silver nitrate solution, which has entered into combination with the formaldehyde, multiplied by 0·3 and divided by the weight of the sample, gives the percentage by weight of absolute formaldehyde. C. A. M.

Analysis of Fulminate Cap-Compositions. Utescher. (*Zeitsch. Schiess- und Sprengstoffwesen*, 1911, **6**, 402.)—Simple mixtures may be separated by treatment with different solvents in succession. A more complicated one, containing, for example, fulminate, potassium chlorate, antimony sulphide, and powdered glass, is dealt with as follows: To 1 grm. of the composition in a 250 c.c. beaker is added 100 c.c. water and 5 c.c. 1 in 5 sulphuric acid, and hydrogen sulphide passed for thirty to forty minutes through the liquid, previously heated to boiling. After cooling, filtering, and washing with hydrogen-sulphide water, the potassium chlorate is estimated in the filtrate by evaporating it in a porcelain basin until all chlorine is driven off, washing into a tared platinum basin, evaporating, igniting until the potassium sulphate begins to fuse, and weighing. To determine the glass, the precipitate, consisting of glass, antimony sulphide, and mercury sulphide, is warmed on the water-bath in the original precipitating vessel with 5 c.c. concentrated hydrochloric acid and 2 c.c. of 50 per cent. tartaric acid, until, with frequent stirring, the fibres of the filter are separated. From a pipette, 1 c.c. concentrated nitric acid (1·4) is added, the liquid heated, stirred, diluted with a small quantity of water, filtered, the filter washed with hot water, dried, ashed in a porcelain crucible, and the glass ignited and weighed. The mercury fulminate is estimated by electrolysis of the filtrate from the glass in a Frary apparatus. A current of 1·5 ampères at 3 volts is passed for two hours, roughened platinum gauze electrodes being used. Good arc-lamp carbons may be employed (two or three times) if preferred, but the platinum is not attacked by the dilute chlorine solution. The electrodes are washed with distilled water into the electrolyte. The cathode is washed with absolute alcohol and ether, and weighed (factor for mercury fulminate, 1·465). The antimony sulphide is determined in the mercury-free electrolyte by neutralising with caustic soda solution (sp. gr. 1·5), adding 5 c.c. more of this solution and 20 c.c. of 10 per cent. sodium sulphide solution (made by decolorising a solution of commercial crystallised sodium sulphide with ammoniacal hydrogen peroxide and filtering), and electrolysing as before, but with a current of 1 ampère, and keeping at 80° to 90° C., washing the cathode in water, moving it about frequently for five minutes in water at 100° C., but not boiling, washing it with distilled water, absolute alcohol, and ether, drying and weighing. To clean the electrodes, mercury may be removed by ignition, and antimony by immersion in 10 parts concentrated nitric acid and 1 part 50 per cent. tartaric acid. In the absence of electrolytic apparatus the filtrate from the glass is boiled, hydrogen sulphide passed through it for thirty to forty minutes, cooled, and the precipitate collected on a tared filter and washed with hydrogen sulphide water. A crucible of 80 c.c. content is placed beneath the funnel, and the antimony sulphide simultaneously dissolved out and washed into it with hot ammonium sulphide solution from a fine jet, followed by hot water. The mercury sulphide left on the filter is washed with hot water, alcohol, ether, and carbon disulphide, dried at 110° C., and weighed. The antimony solution is concentrated on the water-bath, oxidised by evaporation to dryness with nitric acid (1·4) three or four times, and strongly ignited.

If the cap-composition contains organic nitro-compounds, these are extracted first of all with solvents such as benzene. Black powder, the composition of which must be known or found in a separate sample, is estimated by extracting its sulphur with carbon disulphide. If potassium chlorate is simultaneously present, it is estimated with the nitre, or separately in the filtrate from the sulphides of antimony and mercury by driving off hydrogen sulphide, boiling with ferrous sulphate to complete the reduction of the chlorate, acidulating with nitric acid, and precipitating with silver nitrate. O. E. M.

Detection of Small Quantities of Glycuronic Acid. C. Neuberg and S. Saneyoshi. (*Biochem. Zeitsch.*, 1911, 35, 56-59.)—Glycuronic osazone yields a violet coloration soluble in benzene when it is heated with hydrochloric acid and naphtho-resorcinol; the osazones of pentoses, hexoses, and disaccharides, give a similar coloration, but the latter is not extracted from the mixture by benzene. A mixture of the osazones (about 0·008 grm.) is dissolved in 4 c.c. of concentrated hydrochloric acid, an equal volume of water is then added, and the solution is boiled. A quantity of about 0·1 grm. of naphthol-resorcinol is now added, the mixture is heated for thirty seconds, then cooled to a temperature of 50° C., and shaken with benzene. In the presence of glycuronic osazone, a red-violet colour is extracted by the benzene, and the solution exhibits an absorption band in the green-yellow portion of the spectrum. W. P. S.

Estimating Humus in Soils. J. B. Rather. (*J. Ind. Eng. Chem.*, 1911, **3**, 660-662.)—The method of Mooers and Hampton (ANALYST, 1908, **33**, 290) is a great improvement on the American official method, but it does not eliminate as much of the clay as is desirable. In some cases the residue before the final ignition may consist of more than 50 per cent. of clay. Better results are obtained by repeating the extraction with ammonia and evaporation several times; but this occupies much time, and, if acid fumes are present in the laboratory, may introduce appreciable errors. The author recommends the following method, in which the clay is coagulated and thrown out of suspension by means of ammonium carbonate, which can be subsequently eliminated below 100° C. (at about 85° C.). The soil is washed with 1 per cent. hydrochloric acid and water, digested with 4 per cent. ammonia, and filtered as in the official method. In 130 c.c. of the filtrate, 0·65 grm. ammonium carbonate is dissolved, and the solution allowed to stand in a glass-stoppered cylinder overnight. The clear supernatant liquid is then decanted through a dry filter into a dry flask, and 100 c.c. of the filtrate are evaporated in a tared platinum dish, dried for three hours at 100° C., weighed, ignited, and reweighed. The humus (final loss on ignition) is on the average nine-tenths that found by the method of Mooers and Hampton, but with some soils it is less than half as much. Judged by the criterion of Alway, Files and Pinckney (ANALYST, 1910, **35**, 495), the results yielded by the new method are the more trustworthy, as the final ash is always less than that yielded by the method of Mooers and Hampton, and is usually only one-third as great. G. C. J.

Testing of Imitation Parchment Papers for Resistance to Grease. G. Schacht. (*Wochenbl. Papierfab.*, 1911, **42**, 3632-3634; through *J. Soc. Chem. Ind.*, 1911, **30**, 1206.)—For testing "grease-proof" pergamyn or imitation parchment papers, two methods are in use: (1) the paper is required to show blisters when held just above the flame of a burning match; (2) when a little oil of turpentine is rubbed on the surface of the paper it should not penetrate sufficiently to leave a greasy mark on a sheet of white paper spread underneath. A certain amount of controversy has arisen between the advocates of these two methods, each suggesting that the other test is not quite reliable. There appears, however, to be little doubt that certain of these greaseproof papers fail to blister when heated, and yet are perfectly resistant to grease in ordinary use. Whether papers exist which show blisters but are not completely greaseproof is not so certain, but in any case the author considers that the turpentine test should be adopted and the blister test should be discarded. It is a principle in all industrial testing that the test should stand as far as possible in direct relationship to the property to be tested, and for this reason alone the blister test is to be regarded with suspicion. The blister test, if confirmed by the turpentine test, may be useful in the paper machine room as a rapid approximate test for the guidance of the machine foreman, who can if necessary make the adjustments which will increase the blistering properties of the paper when these are deficient. But the turpentine test is the ultimate test for grease resistance, and logically should form the only basis on which this quality is judged.

Candelilla Wax. Niederstadt. (*Chem. Zeit.*, 1911, **35**, 1190.)—Candelilla or canutilla wax is obtained from *Pedilanthus pavonis*, a plant belonging to the *Euphorbiaceæ*, and during the last four years has become a commercial product. Two sorts of Mexican wax are sold, one of which is of lighter colour than the other, and has a hardness similar to that of carnauba wax. Commercial samples of brown and yellow wax gave the following results respectively: Sp. gr. at 15° C., 0·9930 and 0·9360; melting-point, 68·4° C.; solidification-point, 63·8° C.; acid value, 21·13 and 19·16; saponification value, 54·95 and 55·23; and ratio value,* 1·60 and 1·88. This wax is readily soluble in turpentine oil, and is being extensively used for varnishes and for telephone cables. C. A. M.

INORGANIC ANALYSIS.

Modified Cobalt Reaction for the Detection of Very Small Quantities of Hydrogen Peroxide. M. Leuchter. (*Chem. Zeit.*, 1911, **35**, 1111.)—The reagent consists of a mixture in equal proportions of a 1 per cent. solution of crystallised cobalt chloride and of a solution of 1·6 parts borax in 20 parts glycerol (sp. gr. 1·229) and 100 parts water. The test depends on the formation of a dark-coloured ring at the surface of contact of the reagent and the solution to be tested, and the glycerol serves the double purpose of giving the reagent a fairly high density and of preventing the formation of a precipitate when the cobalt and borax solutions are mixed. When 1 to 2 c.c. of a dilute solution of hydrogen peroxide is poured carefully on to an equal bulk of the reagent in a test-tube, a brown zone appears at the surface of contact of the liquids, giving place in time to a finely divided precipitate which at first rises into the upper layer, colouring it brown, but which finally settles. If much hydrogen peroxide be present, there will be a visible evolution of gas. The test serves to detect as little as 0·015 mgrm. of hydrogen peroxide or 0·5 mgrm. of sodium perborate in 1 c.c. Persulphates give no reaction unless either caustic soda is added or the solution is warmed, and the test therefore serves to differentiate between perborates and persulphates (*cf.* Lenz and Richter, Analyst, 1911, 470). G. C. J.

Gravimetric Estimation of Nickel and Cobalt. L. Dede. (*Chem. Zeit.*, 1911, **35**, 1077.)—The precipitation of nickel or cobalt by boiling with alkali and bromine-water is attended with difficulties, the greatest of which is the tendency of the precipitate to retain alkali. This difficulty may be overcome entirely by using potassium (not ammonium) persulphate in place of bromine, and precipitating in the cold. The advantage of persulphate over bromine depends on the fact that, in presence of persulphate, the slightest excess of caustic alkali causes complete precipitation, whereas with bromine a considerable excess of alkali is necessary. The amount of alkali must, of course, be sufficient to neutralise the acid set free on decomposition of the persulphate, and, if ammonium salts are present, they must be first decomposed by boiling with excess of alkali. In absence of ammonium salts, rather more caustic alkali is added to the cold solution than is theoretically required by the nickel or cobalt which may be present, and by the equivalent amount of persulphate. The latter is then added, when, in tolerably concentrated solutions, precipitation is immediate and complete. With very little nickel or cobalt present, a brown solution may result. Except when the nickel and cobalt are mere traces, these brown solutions clear, with complete precipitation of the oxide, if left for an hour or two and shaken from time to time. By allowing to stand overnight, the most dilute solutions deposit their oxide. Precipitation in the cold and the use of cold water for washing secure a precipitate of such a character that any occluded alkali is easily removed by washing. The author reduces the ignited precipitate in a current of hydrogen, and weighs as metal. The extreme error of the method for nickel is ± 0·2 mgrm. on weighings up to 0·3 grm., for cobalt only half as much. G. C. J.

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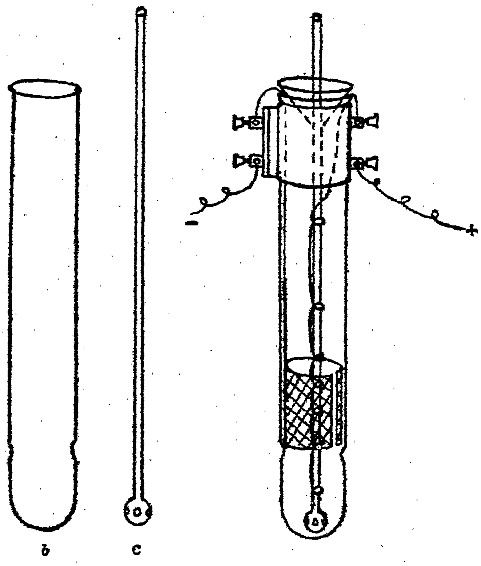
* The ratio between the amounts of alkali required by the free acids and that required to saponify the esters.

Precipitation of Zinc, Manganese, Cobalt, Nickel, Copper, and Cadmium from Ammoniacal Solutions by Means of Sodium Carbonate and Trimethylphenylammonium Carbonate. E. Schirm. (*Chem. Zeit.*, 1911, 35, 1177, 1193-1194.)—The above metals may be precipitated quantitatively from solutions of their complex ammonium salts by boiling with sodium carbonate until the escaping steam no longer smells of ammonia. The method is useful for the separation of zinc as a preliminary to an estimation of sulphuric acid. The zinc is first precipitated in the cold by the addition, drop by drop, of sodium carbonate. The precipitate of zinc carbonate and basic sulphate is next dissolved by addition of just sufficient ammonium carbonate solution and the mixture is heated to boiling. The solution, now cloudy, is cleared by addition of more ammonium carbonate and then boiled until ammonia can no longer be detected in the escaping steam. The precipitate is easily filtered and washed, and no trace of sulphuric acid remains with it. It is, however, never free from sodium, and if it be required to estimate the zinc, must be redissolved and the zinc precipitated as sulphide, or the following modified method may be used :—

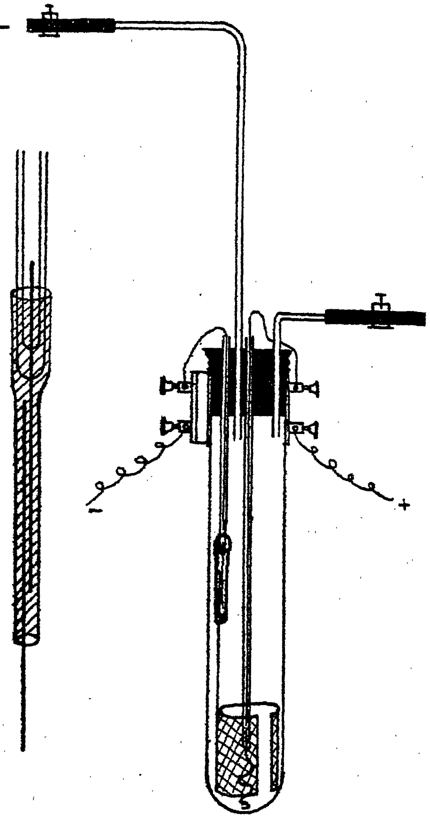
In place of sodium carbonate, trimethylphenylammonium carbonate is used. This is prepared by the interaction of dimethylaniline and methyl iodide and decomposition of the resulting trimethylphenylammonium iodide by rather less than the theoretical amount of moist silver oxide, as the presence of some iodide does not interfere, whilst that of silver would. With this reagent, an accurate estimation of zinc is possible. The precipitate is contaminated by the trimethylphenylammonium carbonate as by sodium carbonate, but this is eliminated on ignition. The precipitate is easily filtered and washed, but, like the precipitate obtained in the sodium carbonate method, sticks to the precipitating beaker. When washing is completed, the beaker is rinsed with hot dilute nitric acid, which is then poured through the filter, the latter washed with hot water, the solution evaporated to dryness, and the residue ignited for ten minutes in a covered crucible over a Bunsen burner and weighed as ZnO. The extreme error of the method is ±0·3 mgrm. with 0·13 grm. zinc oxide present.

When trimethylphenylammonium carbonate is used to separate zinc, any subsequent precipitation of sulphuric acid in the filtrate must be made by the addition of barium chloride drop by drop; the addition at one time of a large quantity of barium chloride gives rise to the formation of a scum, to trouble in washing, and to results which may be 10 per cent. too high. G. C. J.

Rapid Electro-Analysis with Agitation by Means of a Current of Gas. F. Fischer, C. Thiele, and E. Stecher. (*Zeitsch. Electrochem.*, 1911, **17**, 905-906.) —The complete apparatus is shown on the right. The tube *b* is supported by a holder, separately figured in the original paper to show that it consists of a felt-lined collar of non-conducting material, to which are attached two pairs of binding screws and a rod which can be held by the boss of a retort stand. The tube *b* is about 30 cm. × 4·5 cm., and 5 cm. from the bottom it has three indentations which serve to support the cathode. The gas-leading tube *c* is 35 cm. × 5 mm., and has blown on its end a 2·5 cm. bulb, which is pierced by a number of holes, including one at its lowest point. The anode consists of a platinum wire wound round the tube *c*, the windings being comparatively close at the elevation of the cathode. The cathode is a platinum gauze cylinder, open so that it can be dropped into place after the anode wire has been attached to its terminal, and with some 20 cm. of platinum wire welded to the end of the usual stem, so as to connect, as shown, with the binding screw on the left. A stemless funnel dropped over the tube *c*, after the electrical connections are made, serves to keep *c* central. The apparatus has so far been used only with hydrogen as agitating gas. The speed of gas is so regulated that the electrolyte, which normally just covers the cathode, is thrown up some 5 cm. higher. Working at 60° C., there is sufficient condensation of water to wash the walls of the apparatus. From 0·15 to 0·2 grm. of copper or nickel can be deposited in ten minutes with a current of 1·2 amps. (8 volts). The results are exact G. C. J.



Rapid Electro-Analysis under Reduced Pressure. F. Fischer, C. Thiele and E. Stecher. (*Zeitsch. Electrochem.*, 1911, **17**, 906-908.)—Agitation is effected by means of the gas liberated in the course of electrolysis, the volume of this gas being increased a hundredfold by working under diminished pressure. The apparatus is somewhat similar to that described in the preceding abstract, but the tubular container for the electrolyte has no constrictions to support the cathode, and is provided with a rubber stopper. Through this rubber stopper pass four tubes. The long bend is connected to a water-pump throughout the experiment, and at its end is pushed down into the tubular container so that the electrolyte and wash water can be removed by suction. The short bend admits water for washing. A third tube carries the wire anode which is sealed into it at the bottom, the tube being filled with mercury. The fourth tube, also filled with mercury, has a long platinum wire fused through its closed lower end. Over this wire and the end of the tube a piece of capillary rubber tube is passed, and into the rubber tube the stem of the cylindrical gauze anode is passed, thus forming a contact which is easily made and broken. The inset illustration shows this on a larger scale. The apparatus has been used for the estimation of copper in solutions containing 0·1317 grm. in 100 c.c., acidified with 1·5 c.c. sulphuric acid. At a temperature of 70° C., and with 2 to 3 amps. (8 volts), deposition is complete in less than ten minutes. The least accurate of three results given is 0·1314 grm. found with 0·1317 grm. present. G. C. J.



Determination of Dust in Blast Furnace Gas. W. Brady and L. A. Touzalin. (*J. Ind. Eng. Chem.*, 1911, **3**, 662-670.)—Apparatus is figured, and a full description given of the precautions necessary to estimate accurately the suspended matter in a rapidly moving current of gas. The principal conditions which must be observed are as follows: The sample must be withdrawn from that region of the main in which experiment shows that the velocity of the gas is the mean velocity. In withdrawing the sample means must be employed to change the direction of flow gradually (sampling-tube curved with radius of 12 inches), and to cause the flow in the entrance end of the sampling tube to be at the same velocity as the passing stream of gas. When a retardation occurs in the sampling-tube, the distortion of flow round its entrance causes an undue proportion of dust to lodge in the sampling-tube and be carried over into the filter. Giving the gas too high a speed in the sampling-tube, by means of too powerful suction, leads to errors of a contrary kind. These precautions are of the utmost importance where the suspended matter exceeds 1½ grains per cubic foot, and means are described for controlling the speed of gas in the sampling-tube within 2 or 3 per cent. Where possible, samples should be taken from a vertical main, and at least 15 feet from any bend or obstruction. The gas must be filtered above 100° C., and cooled below its dew-point (about 50° C.) before being delivered to the wet test meter. G. C. J.

Determination of Ferrous Iron in Silicate Rocks. M. Dittrich. (*Chem. Zeit.*, 1911, **35**, 1093.)—It is shown that the uncertain end-point in the titration of ferrous iron after decomposition of the rock by means of hydrofluoric and sulphuric acids is unconnected with the presence of manganese, and that the usual precautions fail to make the end-point even reasonably sharp with some rocks. These rocks were in every case found to contain notable proportions of titanium, but titanium dioxide, even in large amount, has no disturbing effect on the titration. Addition of titanous acid, however, to a ferrous solution before titration with permanganate, leads, as might be expected, to exactly the same indefinite end-points which are occasionally observed in the course of rock analysis. It is suggested that rocks may contain titanium as Ti_2O_3, as well as in one or other of the crystalline modifications of the dioxide. This view is confirmed by the fact that sharp end reactions may be obtained by the addition to the titration liquid of excess of finely divided silica and a considerable amount of potassium sulphate. Ferric salts are but slowly reduced by titanous acid in hydrofluosilicic acid solution, and high saline concentrations also tend to retard the reduction. G. C. J.

Volumetric Determination of Lead as Chromate. J. Waddell. (*J. Ind. Eng. Chem.*, 1911, **3**, 638-640.)—The following modification of the method of Guess (*Trans. Amer. Inst. Min. Eng.*, 1905, **35**, 359) is recommended for use with nearly all ores, but is said to be most satisfactory in just those cases where the molybdate method is least trustworthy—namely, with ores poor in lead, and containing much calcium carbonate. The ore (1 to 5 grms.), contained in a 250 c.c. flask, is dissolved in about 15 c.c. of hydrochloric acid, to which 3 to 5 c.c. of nitric acid is subsequently added, and the whole digested on the hot plate until solution is complete and the liquid is reduced to about 8 c.c. At the conclusion of this operation, which requires about fifteen minutes, the flask is removed from the hot plate, and its contents, when cool, are rendered slightly alkaline by addition of ammonia (1 : 1). Acetic acid is then added slowly with vigorous shaking until the liquid smells distinctly of acetic acid. Finally, some 5 c.c. of strong ammonium acetate solution is added to insure complete solution of the lead. If the ore contains no antimony, and no gelatinous silica has separated, any slight silicious residue in the bottom of the flask is disregarded, and the lead is precipitated in the hot, undiluted solution by addition of 10 c.c. of a 10 per cent. solution of potassium chromate. After shaking and allowing to settle for five minutes, the supernatant liquid is decanted off through a filter, and the precipitate in the flask and on the filter is washed with 0·5 per cent. acetic acid containing 0·2 to 0·3 grm. per litre of potassium chromate, and finally with water until free from soluble chromate. The filter is then set over the original flask, and the lead chromate is dissolved by the alternate application to the filter of cold dilute hydrochloric acid (1:3) and cold water, with the occasional use of 5 to 10 c.c. of hot water, following the cold water, the purpose of this procedure being the complete solution of the lead chromate without risk of evolution of chlorine. At least 100 c.c. of the dilute (1:3) acid should be used, as the accuracy of the final titration depends on the presence of a sufficient quantity of acid. To the hydrochloric acid solution of the lead chromate, about 1 grm. of potassium iodide is added, and then standard thiosulphate is run in from a burette at the rate of about 3 drops a second, using starch towards the end of the titration. The amount of potassium iodide used should not much exceed that stated, nor should the solution be specially cooled before titration, since low temperatures and excess of iodide give rise to the formation of yellow scales of lead iodide, which tend to obscure the end-point. The extreme error of the method is ± 0·1 c.c. of standard solution which, with the quantities taken for analysis by the author, corresponds to ± 0·3 per cent. of the lead present. Antimony, bismuth, silver, and gelatinous silica interfere, and, with large quantities (say, 20 per cent.) of iron in the ore, the tendency to form basic acetate causes difficulty in dissolving the lead. G. C. J.

Use of Logwood in Qualitative Analysis. Estimation of Bismuth. E. Vassallo. (*Gazz. Chim. Ital.*, 1911, **41**, 204-212.)—Logwood paper, prepared by saturating filter paper with an extract of 50 grms. of powdered logwood in 100 grms. of alcohol, gives characteristic reactions with solutions of different metals. Salts of bismuth give an intense violet coloration, which is stable even in an acid solution. Arsenic, antimony, and tin also give a violet colour in a neutral solution, but when sufficient acid is present only a red colour, due to the acid, is obtained; whilst molybdic solutions give an intense blue coloration. In applying the test it is best to allow the drop of liquid under examination to fall upon the test-paper, which is floated upon water slightly acidified. For the rapid detection of tin or bismuth in a mineral or alloy, the powdered sample is treated with nitric acid and a few crystals of sodium sulphate (to prevent the formation of insoluble bismuth arsenate), and heated until nitrous fumes appear. The residue is taken up with water, and the liquid filtered from the insoluble hydroxides of antimony and tin, whilst any bismuth arsenic, and molybdenum will be in the filtrate. The contents of the filter are washed with water acidified with nitric acid, and boiled with a saturated solution of tartaric acid, which will dissolve the antimony and leave the tin hydroxide. The latter, when boiled with a little sodium hydroxide and water, will give a solution, which will colour the logwood paper blue on account of the alkali present; but when the paper is floated on water acidified with nitric acid, the alkali is neutralised, and a violet coloration due to the tin will appear. The filtrate from the tin and antimony must be freed from molybdic acid by treatment with ammonia and sodium phosphate before testing for bismuth with the logwood paper. The test may be used in the volumetric estimation of bismuth, a standardised solution of disodium phosphate being added little by little to a dilute solution of bismuth nitrate which has been neutralised with ammonia, and then rendered acid again with 1 drop of dilute nitric acid. It is essential that the drops for spotting the test-paper should not be taken until the successive precipitates formed in the titration have completely subsided. The tabulated results thus obtained vary from + 0·11 to + 0·34 per cent. from the theoretical amounts of bismuth. C. A. M.

Estimation of Magnesium as Oxide. O. Kallauner. (*Chem. Zeit.*, 1911, **35**, 1165-1166.)—Whether ignited alone or in presence of mercuric oxide, the conversion of magnesium chloride to oxide is never quantitative. The proposal of Pfeifer (*Zeitsch. angew. Chem.*, 1909, **22**, 435), to heat to 450° C. only, leads to grave errors. By the use of a much higher temperature—namely, the highest attainable by means of a Teclu burner, or by double treatment with mercuric oxide, an hour suffices to give results sufficiently accurate for some technical purposes. By this the author means with an error not exceeding 1 in 400. G. C. J.

Estimation of Manganese in Vanadium and Chrome-Vanadium Steels. J. R. Cain. (*J. Ind. Eng. Chem.*, 1911, **3**, 630-631.)—Watters (*Met. Chem. Eng.*, 1911, **9**, 244) has described a method for the estimation of manganese in steels containing chromium and tungsten which eliminates the errors introduced by the use of the unmodified bismuthate method in the analysis of such steels. The steel is dissolved in sulphuric acid and oxidised by means of nitric acid, the solution is nearly neutralised, and the chromium and iron are precipitated by addition of an emulsion of zinc oxide. The mixture is made up to known bulk and filtered and in an aliquot portion of the filtrate manganese is estimated by the bismuthate method. The author has found the method satisfactory even with steels containing 0·2 per cent. of vanadium, but he suggests that for high vanadium products his cadmium carbonate method of precipitating chromium and vanadium (ANALYST, 1911, 476) is to be preferred. As a preliminary to a manganese determination, this method is carried out as follows: The steel (1 to 2 grms.) is dissolved in sulphuric acid (1 : 10), observing the precautions given in the paper quoted, and, without oxidising the iron, cadmium carbonate is used in the manner already described to precipitate the chromium and vanadium. Provided the iron is kept mainly in the ferrous state, no manganese will be co-precipitated. The solution is filtered, boiled with 25 c.c. of nitric acid until free from fumes, cooled and used for the estimation of manganese by the bismuthate method. G. C. J.

New Method of Separating Iron from Manganese. J. A. Sanchez. (*Bull. Soc. Chim.*, 1911, [iv.], **9**, 880-881.)—The hot solution of ferric and manganese salts is treated with pyridine, which precipitates the iron quantitatively as hydroxide, leaving the manganese in solution. In this way it is possible to separate 0·005 grm. of manganese from 1 grm. of iron. For the analysis of manganese ores 1 grm. of the sample is decomposed with hydrochloric acid, the silica separated in the usual way, and the bulk of the acid in the filtrate and washings neutralised with sodium hydroxide solution. The liquid is now made up to 200 c.c., treated with pyridine, and boiled for ten minutes, after which the precipitated iron hydroxide is separated and washed first with hot water saturated with pyridine and then with boiling water. When much manganese is present, the iron should be redissolved in hydrochloric acid and the precipitation repeated. In the case of minerals containing metals of the first group, the solution is treated with hydrogen sulphide, and the filtrate oxidised, nearly neutralised, and treated with pyridine as described. Zinc, if present, is also partially precipitated. In that case the precipitate should be redissolved in hydrochloric acid, and the iron reprecipitated by ammonium chloride and ammonia. C. A. M.

Analysis of Calcium Hypophosphite. E. Rupp and Kroll. (*Arch. Pharm.*, 1911, **249**, 493-497.)—A method of analysing calcium hypophosphite is based upon its quantitative oxidation to phosphate by means of bromine. The reagent contains 3·35 grms. of potassium bromate and 15 grms. of potassium bromide per litre, and 28 c.c. of it should liberate from acidified potassium iodide solution an amount of iodine corresponding to about 30 c.c. of $\frac{N}{10}$ thiosulphate solution. In making an estimation, 1·5 grms. of the calcium hypophosphite are dissolved in 500 c.c. of water, and 10 c.c. of the solution shaken in a stoppered flask with 50 c.c. of the reagent, after which 10 c.c. of dilute sulphuric acid are introduced, and the flask left for an hour in a dark place. Potassium iodide (1 grm.) is now introduced, the flask shaken, and allowed to stand for one or two minutes, and the liberated iodine titrated with $\frac{N}{10}$ thiosulphate solution; 1 c.c. of thiosulphate is equivalent to 0·00212 grm. of calcium hypophosphite. C. A. M.

Improved Hollemann's Method for the Estimation of Reverted Phosphoric Acid. E. Vittoria. (*Chem. Zeit.*, 1911, **34**, 1152.)—When a superphosphate is boiled for a few hours in a cold-saturated solution of ammonium nitrate, almost the whole of the phosphoric acid goes into solution. About 2 grms. of the superphosphate, finely ground and dried at 115° to 120° C., are vigorously boiled for about ten hours with 100 to 150 c.c. of ammonium nitrate solution, 10 c.c. of sodium acetate solution, and 500 c.c. of water; the water lost by evaporation is replaced from time to time, and the liquid is finally made up to 1 litre. Forty c.c. of this solution are made slightly alkaline with sodium hydroxide and shaken with 20 c.c. of $\frac{N}{10}$ silver nitrate solution; the silver phosphate is filtered off rapidly, and the excess of silver in the filtrate is titrated according to Volhard's method. J. F. B.

Assay of Platinum. A. Steinmann. (*Schweiz. Wochsch. Chem. u. Pharm.*, 1911, **49**, 441-444, 453-457; through *Chem. Zentralbl.*, 1911, II., 1061-1062.)—The ordinary assay for platinum consists of cupellation of the metal with lead, and at least twice its weight of silver, followed by rolling out the button, and parting the silver by means of boiling concentrated sulphuric acid. It is usual to run a control assay on platinum of known fineness, but it is said that the amount of platinum dissolved by the concentrated acid is very variable. It averages 2 to 3 per cent. of the metal, but may reach 5 per cent. The dissolved platinum may be recovered by evaporation of the parting acid, treatment of the residue with *aqua regia* to separate silver, and separation of the platinum finally as ammonium platinichloride. But the following modified method of assay is preferred. It depends mainly on the fact that sulphuric acid of 85 per cent. strength does not dissolve any appreciable quantity of platinum from its alloys with silver, whilst dissolving the silver readily.

The platinum or alloy is cupelled with silver, copper, and lead, the copper being approximately equal to the platinum present, the silver at least five times as much, and the lead according to a scale given in the paper. The button is rolled out to a thickness not exceeding 0·2 mm., and parted by the use of slightly diluted sulphuric acid (5:1), taking care that the temperature does not exceed 250° C. At 240° C. parting is complete in fifteen minutes. G. C. J.

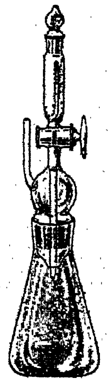
Detection of Small Quantities of Potassium as Cobalti-Nitrite. L. T. Bowser. (*J. Amer. Chem. Soc.*, 1911, **33**, 1566-1569.)—It has been found that by the method previously proposed (ANALYST, 1910, **35**, 141) the presence of alcohol with the mixture of potassium salt and cobalti-nitrite solution not only shortens the time of precipitation but increases the sensitiveness of the test some ten times. The omission of acetic acid, formerly employed, gives better results, the method being modified as follows: Five c.c. of the potassium solution in a tall beaker of narrow base and 50 or 100 c.c. capacity, and 5 c.c. of water in a similar beaker, are placed on a mirror, and 2·5 c.c. of sodium cobalti-nitrite reagent and 5 c.c. of alcohol (95 per cent.) added to each, rotating the solutions to mix the reagents. If upon looking downwards through the solutions into the mirror an equally sharp image of the observer can be seen in each beaker, potassium is absent; in the presence of two or more parts per million of this element the test solution yields a dimmer image than the blank test. It is necessary to avoid the presence of ammoniacal fumes and the presence of ammonium salts in the solution, and to have the solution neutral or slightly acid with acetic acid. Metallic salts do not interfere with the test. The solutions should be compared within a minute or two of mixing the reagents, as the first-formed precipitate gradually disappears. A. R. T.

Rapid and Accurate Method for the Analysis of White Metals. J. C. Beneker. (*J. Ind. Eng. Chem.*, 1911, **3**, 637-638.)—Tin and antimony are separated from other metals by a modification of Rössing's method (*Zeitsch. anal. Chem.*, 1902, **41**, 1), and their separate estimation depends on the fact that, in acid solution, both stannic and stannous sulphide yield stannic iodide on treatment with iodine, whilst both antimonious and antimonic sulphide yield antimonious iodide, the whole of the sulphur separating as such if iodine be present in excess. The antimonious iodide is subsequently titrated with iodine in presence of excess of sodium bicarbonate and the tin calculated from the volume of iodine solution required in this titration and that consumed by the mixed sulphides. The drillings (0·5 grm.) are dissolved in 25 c.c. of hydrochloric acid and 5 c.c. of nitric acid or in 25 c.c. of hydrochloric acid saturated with bromine. Tartaric acid (1 grm.) is added, and the mixture warmed on the steam bath. After diluting to about 400 c.c., the solution is made alkaline with caustic soda, and 20 c.c. of 10 per cent. sodium sulphide is then added. The sulphides of copper, lead, iron and zinc are washed twice by decantation, and subsequently on a filter with a hot dilute solution of sodium sulphide. The filtrate is rendered just distinctly acid with hydrochloric acid, and kept on the steam bath for an hour. The sulphides of tin and antimony (and arsenic if present) are then filtered off and washed with lukewarm water until free from hydrogen sulphide. They are next transferred with the filter-paper to a conical flask. A measured excess of $\frac{N}{10}$ iodine solution, 30 to 50 c.c. of hydrochloric acid and 2 grms. of tartaric acid are added to the contents of the flask, which is then fitted with a rubber bung carrying a swan-neck thistle tube containing a solution of potassium iodide. The contents of the flask are heated to boiling for a few minutes, then cooled and titrated with $\frac{N}{10}$ thiosulphate.

The titrated mixture is washed (or filtered if a sharper end point is desired) into a beaker, neutralised with sodium bicarbonate, after addition of powdered sodium bicarbonate, the antimonious iodide is titrated with $\frac{N}{10}$ iodine. One and a half times the volume of iodine required in this final titration is subtracted from that consumed by the mixed sulphides, and the remainder is calculated to tin (Sn = 4I). The results for antimony may be as much too low as corresponds to 0·1 c.c. of $\frac{N}{10}$ iodine, whilst those for tin may be as much too high as corresponds to 0·2 c.c. $\frac{N}{10}$ iodine, but are never below the truth. G. C. J.

Separation of Zinc and Sulphuric Acid. E. Schirm. (*Chem. Zeit.*, 1911, **35**, 1177, 1193-1194.)—*See* Precipitation of Zinc, etc., from Ammoniacal Solution by Means of Sodium Carbonate and Trimethylphenylammonium Carbonate (ANALYST, 1911, 599). G. C. J.

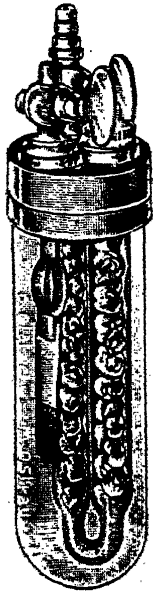
APPARATUS, ETC.



Apparatus for the Estimation of the Hexabromide Value of Oils. C. Niegermann. (*Chem. Zeit.*, 1911, **34**, 1131.)—The apparatus comprises a strong-walled Erlenmeyer flask of 200 c.c. capacity into which is ground a stoppered dropping funnel to contain the bromine; a side-tube is also provided in the stopper to allow the air to escape. For the estimation of the hexabromide value according to Hehner and Mitchell, which value stands in definite relationship to the iodine value of the oil, 1 to 2 grms. of the sample are weighed out into the flask and dissolved in about 40 c.c. of ether and 5 c.c. of concentrated acetic acid. The solution is thoroughly cooled, and bromine is admitted drop by drop, care being taken to keep the flask quite cold, until the brownish-red coloration is permanent. The liquid is allowed to remain for three hours, and the bromide is then collected on a tared filter dried at 100° C. The precipitate is washed with a few c.c. of cooled acetic acid, a little alcohol, and lastly with ether; it is then dried in the water-oven to constant weight. The resistance of the filter-paper to dilute bromine solution is controlled by a blank experiment. Swedish filter-paper is recommended in preference to asbestos. One dropping funnel and stopper may be made to fit several flasks. J. F. B.

Prevention of Bumping. H. Spurrier. (*J. Amer. Chem. Soc.*, 1911, **33**, 1632-1633.)—The author introduces into the flask containing the solution to be boiled a glass tube, open at both ends, and of such a length that the upper end rests, out of the liquid, against the side of the vessel. In the case of a liquid in a beaker, a cover should also be used to prevent loss. The device is particularly useful in the evaporation of the alcohol from aqueous liquids after saponification of an oil, where an oily layer remains on the surface of the liquid.

A. R. T.



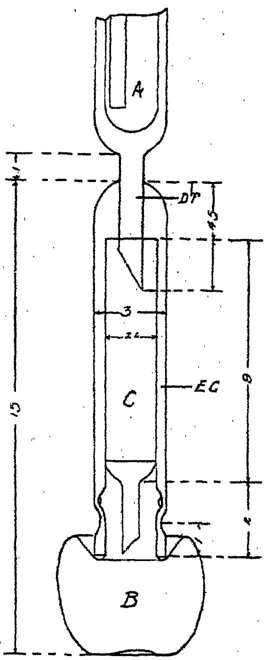
Carbon Monoxide Detector. R. Nowicki. (*Chem. Zeit.*, 1911, **34**, 1120.)—An apparatus by which even the smallest traces of carbon monoxide may be detected in the air is illustrated. It consists of a glass vessel strengthened by metal bands and fitted with a tap for the introduction of the air to be tested. A rubber bellows is attached to this tap, and is operated until the air passes through the vessel and out at a second tap. This second tap carries a clip, from which is suspended a strip of test-paper sensitive to carbon monoxide. The strip of paper is moistened with palladium chloride solution and becomes brown or black if any carbon monoxide be present. The time required for the appearance of the reaction is a measure of the proportion of carbon monoxide in the air, as ascertained by reference to a table. For instance, the presence of 0·01 per cent. by volume of carbon monoxide will give a visible change on the test-paper after eleven minutes, and total blackening after sixty minutes. The instrument is intended to be used for detecting escapes of gas from pipes or furnaces.

J. F. B.

New Accelerated Test for Paints. H. K. Benson and C. Pollock. (*J. Ind. Eng. Chem.*, 1911, **3**, 670-671.)—The bubbling test of Cushman (*Bull.* 35, *Office of Public Roads, U.S. Dept. of Agriculture*) for passivity of painted iron surfaces is useful, but is not sufficient alone to determine the efficiency of a paint. For example, its use alone places aluminium paint above all others in efficiency, which is not in accord with practical experience. The use of the following additional test is recommended: It depends on the measurement of the average electrical conductivity of a film of the paint during a six hours' test under standard conditions. Test pieces of steel, 1·4 × 1 × 0·3 inches, are prepared, and should preferably have rounded edges, as this facilitates rapid and uniform drying of the paint. Before painting, each test piece is put in good metallic connection with a stout copper wire. This is easily effected by bending of the wire at right angles half an inch from its end, and wedging the bent portion into a notch cut into the test piece with a hack saw. When these wires have been attached, the test pieces are given two coats of paint at intervals of a week, and when the second coat is apparently dry are kept for twelve hours in an oven at 45° C. Each test piece is then made the anode in a cell containing a normal salt solution, the cathode of platinum being placed at a distance of 4 inches. The copper conducting wire and the cathode are connected to a source of constant E.M.F.

(8 volts), a milliammeter being included in the circuit. Readings are taken immediately, and again at the expiration of ten, twenty, and thirty minutes, and one, two, and six hours, from the commencement of the experiment. The final readings are of the order of 100 milliampères. The reciprocal of the average current passing during the test is the measure of the resistant or insulating value of the paint. It is suggested that the product obtained by multiplying this value by the inhibitive value, as determined by Cushman's test (*loc. cit.*), is a better measure of the practical efficiency of a paint than either of these values alone. G. C. J.

Extraction Apparatus. C. K. Francis. (*J. Ind. Eng. Chem.*, 1911, 3, 673-674.)—Sealed on to a condenser, the lower end of which is shown in the illustration, is a tube, EC, 3 c.m. in diameter and constricted laterally to 2·7 c.m., at a point about 1 c.m. from its base which dips into the mercury contained in the re-entrant angle formed by the shoulder of the modified Knorr flask. The constriction runs nearly but not quite round the tube, so that in one position the points blown out on the neck of the flask can pass up into the tube EC. By giving the flask a quarter turn it is then supported by the points which rest on the constriction, thus dispensing with the necessity of any special support for the flask. The points on the neck of the flask are blown right out so as to provide two holes about 3 mm. in diameter for the upward escape of vapour to the condenser. The neck of the flask ends at the horizontal line below the letter C. The extraction tube C rests on the neck of the flask and is packed with cotton, asbestos, etc., before introducing the sample, or it may be replaced by a paper thimble supported on a very small funnel. G. C. J.



Rapid Filtering Funnel. H. Spurrier. (*J. Amer. Chem. Soc.*, 1911, **33**, 1584-1585.)—A funnel of the ordinary pattern filters far more rapidly if a series of etched lines (six or eight in number) be made from a point a little below the apex to a distance about halfway up the sides. A. R. T.

Consistency of Paints by the Stormer Viscosimeter. A. Rogers and A. H. Sabin. (*J. Ind. Eng. Chem.*, 1911, **3**, 737-738.)—The authors describe the use of the Stormer viscosimeter fitted with the cylindrical modification suggested by Forrest, as applied for the testing of paints. Glycerol of 95 per cent. strength is adopted as the standard, at a temperature of 20° C. The viscosimeter consists of a flat-bottomed cylindrical cup, $1\frac{7}{8}$ inches internal diameter by $2\frac{1}{2}$ inches deep, having two radial vertical wings or septa, $\frac{1}{4}$ inch wide, attached to its inner wall and extending from the bottom to within $\frac{5}{8}$ inch from the top, being obstructions to the rotary flow of the liquid. Within the cup is suspended a stirrer, the revolution of which is resisted by the viscosity of the liquid; this stirrer is a hollow metal cylinder $1\frac{3}{8}$ inches long and $1\frac{1}{4}$ inches in diameter, its lower end open, the upper end closed except for four holes, each $\frac{3}{16}$ inch in diameter, arranged symmetrically. The cylinder is supported from above by a vertical rod or shaft concentric with the cylinder, but external to it, being $\frac{3}{16}$ inch in diameter by $2\frac{1}{4}$ inches long. The end of the shaft is attached by a collar and screw to the bottom of a vertical shaft carrying a pinion $\frac{1}{2}$ inch in diameter with 25 teeth driven by a gear $5\frac{1}{4}$ inches in diameter with 275 teeth, to which is attached a drum around which is wound a small silk cord passing over a pulley and carrying a driving weight. The top of the shaft to which the small gear is attached also carries a worm, which engages and drives the serrated edge of a graduated disc, one revolution of which marks 100 revolutions of the stirrer. The stirrer is suspended so that its bottom is $\frac{3}{8}$ inch above the bottom of the cup, and its top is $\frac{3}{4}$ inch below the top of the cup; when in use the liquid stands $\frac{3}{8}$ inch above the top of the stirrer. To make 100 revolutions of the stirrer the weight descends about 39 inches. There is a clearance of $\frac{1}{16}$ inch between the stirrer and the two radial wings projecting from opposite sides of the cup.

The viscosity of any liquid placed in the cup is measured by the number of grms. required to cause the stirrer to make 100 revolutions in one minute; allowance must be made for the friction of the machine, which in a particular case was 5 grms. The instrument is used with a stop-watch, and is fitted with a friction clutch which, being opened, allows it to start. It is very important to make all observations at a standard temperature. J. F. B.

Substitute for Rubber Stoppers. (*Chem. Zeit.*, 1911, 35, 596.)—The device consists of a cap of rubber, with a hole bored in the crown for the reception of a leading tube. The cylindrical part of the cap, which is stretched over the neck of the flask, is of moderately thin rubber tube, but the crown is of sufficient thickness to give a reasonably long bearing to the tube which penetrates it, and thus to mainlain this approximately vertical. The caps are made by Bernard Tolmacz and Co., Berlin. G. C. J.

ERRATUM.—Page 526, lines 11 and 12, should read: "After washing free from sodium platinichloride and excess of hydrogen platinichloride, the potassium salt is dissolved. . . ."

* * * * *

REVIEW.

Analytical Chemistry. By F. P. Treadwell, Ph.D., Professor of Analytical Chemistry in the Polytechnic Institute of Zurich. English Edition, translated from the German by W. T. Hall, S.B., Instructor in Chemistry, Massachusetts Institute of Technology. Vol. II., Quantitative Analysis. Second Edition, thoroughly revised and enlarged. New York: John Wiley & Sons. London: Chapman & Hall. 1910. Pp. 787. Price $4.00.

This well-known work has already passed through four editions in German, which fact in itself constitutes a proof of its appreciation. The volume under review is the second English edition, and the text having been compared by the translator with the fourth German edition, certain additions have been made which are not to be found in the latter. Indeed, Mr. Hall, by reason of the numerous notes he has added, is rightly to be regarded as joint author.

Although there is no preface, it is plain that the work is intended as a students' textbook, and as such it fulfils its object, whilst it will also prove extremely valuable to the practising chemist, though obviously from its compass it cannot claim to be an exhaustive treatise. Such a work, in which the subject of quantitative analysis is dealt with on scientific lines, has long been needed for tutorial purposes, and the present volume certainly supplies this want.

The introductory chapter is concerned with generalities, and it contains some useful information on the principles of the balance, filtration and washing of precipitates, evaporation of liquids, drying of substances, etc. In regard to the washing of precipitates, the authors give the well-known formula for calculating the quantity of water required for this purpose; but although they point out that some precipitates require more water for their washing than others, and attribute this to adsorption, they omit to warn the student that it is not always advisable to drain a precipitate as dry as possible before the addition of washing water. That this is the case every practitioner is aware; nor, may it be added, is it invariably confined to amorphous, gelatinous precipitates. The ignition of wet precipitates is widely adopted by chemists, and there are cases where it is actually desirable; yet by most teachers it has hitherto been regarded as a rough-and-ready method. It is therefore interesting and satisfactory to observe that the present authors recommend it for those precipitates which do not suffer any permanent change by the combustion of the filter paper.

Following the introduction there are three main parts devoted to gravimetric analysis, volumetric analysis, and gas analysis, each part being subdivided. The exceptional order in which the gravimetric estimation of the metals is described appears difficult to justify. Thus, commencing with the alkali metals, the text deals *seriatim* with the alkali earths, the iron group and the sulphuretted hydrogen group. The last-mentioned group is further subdivided into "sulpho bases," comprising mercury, lead, bismuth, etc., and "sulpho acids," comprising arsenic, antimony, tin, gold, etc. This nomenclature will probably offend the susceptibilities of some of our purist colleagues. Non-metallic elements are classed together collectively as metalloids!

The methods both gravimetric and volumetric given throughout the book are for the most part well chosen, the descriptions are clear and concise, whilst the whole spirit of the teaching is based on scientific principles. The estimation of organic compounds in general does not enter into the text, although Kjeldahl's nitrogen method is described under ammonium. Here it is disappointing to find that potassium permanganate is still mentioned as one of the substances to be added to the sulphuric acid with which the substance being analysed is heated. Again the Jodlbauer modification is simply referred to, whereas a description of the latest modification should certainly have been included. The ninety pages of the text devoted to gas analysis contain some valuable matter for the student.

The text is well printed and the descriptions of the methods are rendered clearer by numerous illustrations. The book is distinctly to be regarded as a welcome addition to the literature of the subject. ARTHUR R. LING.

* * * * *

NEW BOOK.

THE MANUFACTURE OF CHEMICAL MANURES. By J. FRITSCH. Translated from the French, with numerous notes, by DONALD GRANT. Scott, Greenwood and Son, London, 1911. Price 10s. 6d. net.

This octavo volume contains 332 pages of subject-matter, with 69 illustrations, 108 tables, and an alphabetical index of subjects. The classification of the test is as follows: I. Phosphoric Acid. II. Principal Phosphate Deposits. III. Drying and Enrichment of Phosphates. IV. Historical Review of Superphosphate Manufacture. V. Theory of Manufacture of Soluble Phosphates. VI. Manufacture of Superphosphates. VII. Crushing, Sifting, Drying, and Storing of Superphosphate. Retrogradation. VIII. Compound Manures. IX. Manufacture of phosphoric acid, Double Superphosphates, and Various Products. Manufacture of Phosphorus in the Electric Furnace (Appendix). X. Manufacture of Bone-Dust and of Bone Superphosphate (Vitriolised Bones). XI. Basic Slag. XII. Nitrogenous Manures. XIII. Manure from Animal Waste. XIV. Recovery of Nitrogen from Distillery Spent Wash. Cyanamide and Nitrate of Lime. XV. Nitrogenised Phosphatic Manures. XVI. Potassic Manures. XVII. Transference and Handling of Raw Materials and Finished Products.

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INDEX TO VOLUME XXXVII.

INDEX OF NAMES.

ABDERHALDEN, E., *and* Hanslian, R., Ester Method for the Detection of Mono-Amino-Acids together with Polypeptides, 272
————, *and* Kautzsch, K., Esterification of Monamino Acids by Means of Ethyl Iodide, Separation of Pyrrolidone-carboxylic Acid from Glutamic Acid, 360
Achert, O., Inversion of Sucrose by the Enzymes of Honey, 194
Agnew, J. W., *and* Croad, R. B., The Constituents of Oil of Savin, 295
Ahrens, C., Examination of Storax, 499
Aleixandre, P., Microchemical Reaction of Spermatic Fluid, 358
Allen, J. C., *and* Jacobs, W. A., Determination of Water in Petroleum and its Products, 573
————, Physical and Chemical Properties of the Petroleums of the San Joaquin Valley of California, 104
——, W. S., Rational Analysis of Nitrate of Soda, 523
————, *and* Bishop, H. B., Exact Determination of Sulphur in Pyrites, 524
————, *and* Palmer, R. M., Determination of Small Quantities of Arsenic by the Gutzeit Test, 579
Alpers, K., Refractometer Value of the Serum of Cow's and Goat's Milk, 317
Andersen, A. C., Use of Charcoal as a Clarifying Agent in the Estimation of Sugar in Urine, 57
Andrews, E. R., Coste, J. H., *and* Shelbourn, E. T., The Examination of Mixtures containing Petroleum, 8
Anneler, E., Estimation of Morphine in Opiates, 308
Archbutt, L., An Apparatus for Testing Water by Measurement of its Electrical Conductivity, 538
————, On the Application of Adsorption to the Detection and Separation of Certain Dyes, 344
————, The Detection of Heavy Petroleum in Paints and Vegetable Oils, 249
————, The Analysis of Lithopone, 241
————, The Estimation of Ammonia in Carbonated Waters, 177
————, The Examination of Chinese and Japanese Wood Oil, 553
————, The Examination of Mixtures Containing Petroleum, 12
————, The Phosphomolybdate Estimation of Phosphoric Acid in Soils, 136
————, The Separation of Arsenic from Antimony and Other Metals, with Some Applications to Toxicological Work, 237
—, The Souring of Milk, 172
Arnaud, F. W. F., *and* Hawley, H., Notes on the Determination of Butter Fat and Cocoanut Oil in Margarine, 122
Arnold, H., Analysis of Platinum Alloys, 420
————, W., Estimation of the Molecular Weight of Small Quantities of Fatty Acids, 203
————, Mkonga Oil, 256
————, Sources of Error in the Estimation of the Polenske Value of Fats, 256

Astruc, A., *and* Jadin, F., Amounts of Arsenic in Vegetable Foods, 254

Atz, —, Influence of Lucidol (Benzoyl Peroxide) on Certain Qualitative Tests for Oils, 273

Auld, S. J. M., An Introduction to Quantitative Analysis, *Review*, 337

————, The Phosphomolybdate Estimation of Phosphoric Acid in Soils, 130

Aumann, —, Value of Direct Enumeration of Bacteria in Water by Means of the Ultra-Microscope, 317

Austin, W. L., *and* Keane, C. A., The Analysis of Lithopone, 238

d'Auzay, P. J., *and* Bruno, A., Estimation of Sulphates in Solution by the Physico-Chemical Volumetric Method, 282

Bach, H., Colorimetric Estimation of Phenols in Effluents, 39

Bachmann, W., Zsigmondy, R., *and* Stevenson, E. F., Apparatus for Determination of Vapour Pressure Isothermals of Silicic Acid Gels, 328

Bacon, R. F., *and* Dunbar, P. B., Apparatus for Extracting Liquids with Immiscible Solvents, and for Measurement of Evolved Gases, 69

————————, Determination of Malic Acid, 29

———, W., Estimation of Furfural by Means of Fehling's Solution, 46

————, Note on a New Preservative for Milk, Cream, etc., 178

————, On the Relation between the Kirschner and Polenske Values for Margarines Containing Cocoanut or Palm Kernel Oils, 188

Baessler, O., Rapid Estimation of Pyridine in the Presence of Ammonia in Gas-Water, 574

Bailly, O., Estimation of the Amino-Acids in Plants by Means of the Formaldehyde Method, 312

Baker, J. L., *and* Day, F. E., The Iodimetric Titration of Sulphites in Presence of Alcohol and Sugars, 439

Baldoni, A., Quantitative Estimation of Quinine in Urine and in Blood, 455

Bannister, C. O., *and* McNamara, W., On the Effect of Lime on the Ammonium Molybdate Method of Lead Assay, 242

Bardach, F., *and* Ditz, H., Estimation of Phenol and *p*-Cresol in the Presence of Each Other, 62

Barnebey, O. L., Rare Earth Reactions in Non-Aqueous Solvents, 524

Baskerville, C., *and* Crozier, W. J., Determination of Ozone by Means of Cadmium Potassium Iodide, 587

Bateman, E., Modification of the Sulphonation Test for Creosote, 269

Batey, J. P., *and* Knecht, E., Modification of the Beckmann Apparatus, 384

Baubigny, H., Detection of Bromine by Fluoresceïn, 145

Baudisch, O., *and* Coert, J. H., Angeli's Reaction for Aldehydes, 359

Baumann, P., Use of the Mercury Cathode, Particularly in the Electro-Analytical Separation of Metals, 279

Baumert, G., Polarimetric Estimation of Banana Starch, 560

Bayer, A., Quantitative Separation and Estimation of Copper by Means of Hydroxylamine Hydrochloride, 583

————, Separation and Estimation of Pyridine and Ammonia, 413

Beadle, C., *and* Stevens, H. P., A Method for Determining the Amount of Insoluble Particles in Raw Rubber, 13

Beam, W., Mechanical Analysis of Arid Soils, 588

Becaia, M., *and* Mélikoff, P., Estimation of Phosphoric Acid in Presence of Colloidal Silicic Acid, 214

Becker, R., Hübener Bromide Method for the Estimation of Caoutchouc, 509

Beckmann, E., Porcelain Bunsen Burners, 426

————, Sodium Flame Lamps for Polarisation, 594

Beckmann, E., *and* Weber, W., Ebullioscopic Determinations at Low Temperatures (−36° to −83° C.), 287

Behre, A., Alteration of the Acid-Content of Distilled Vinegar when Stored in Wooden Casks, 23

———, Detection of Saponin and Glycyrrhizin in Lemonades by Means of Vamvakas' and Frehse's Tests, 22

———, *and* Frerichs, K., Fruit Juice Statistics for the Year 1911, 94

Beis, C., New Method for the Estimation of Glycerol in Wines, 351

Beistle, C. P., Exudation of Nitroglycerine from Dynamite, 572

Beltzer, J. G., *and* Persoz, J., Les Matières Cellulosiques, *Review*, 74

Benner, R. C., *and* Ross, W. H., Filtration with Alundum Plates, 109

Bennett, R. R., Solubility of Ether in Normal Salt Solution, 448

Berberich, F. M., Burr, A., *and* Wolff, A., Parchment Paper, 465

Berczeller, L., Estimation of Fat and Lipoids in Blood, and Notes on the So-Called Lipolysis, 561

Berkhout, J. D., Estimation of Mercury in Smokeless Powder, 273

Berlin, E., Two New Pieces of Laboratory Apparatus, 528

Bernardi, A., Influence of Peptones on the Estimation of Reducing Sugars by Fehling's Solution, 367

Besson, A. A., Analysis of Lactic Acid, 29

Betti, M., Method of Distinguishing between Aldoses and Ketoses, 266

Bevan, E. J., On the Application of Adsorption to the Detection and Separation of Certain Dyes, 343

———, *and* Cross, C. F., Estimation of Cellulose in Woods and Textile Fibres, 510

Beyne, E., Estimation of Zinc in Ferruginous Minerals, 425

Bidtel, E., Valuation of Fluorspar, 210

Biernath, O., Detection of Benzoic Acid in Foods, 349

Bishop, H. B., *and* Allen, W. S., Exact Determination of Sulphur in Pyrites, 524

Björn-Andersen, H., Estimation of Dry Matter in Potatoes and the Applicability of the Ordinary Tables, 259

Black, J. W., Cockburn, J., *and* Gardiner, A. D., The Gravimetric Separation of Zinc and Nickel, 443

Bleyer, B., *and* Boshart, K., Gravimetric Estimation of Beryllium, 581

———, *and* Moormann, A., Volumetric Estimation of Beryllium, 276

Blockley, J. R., *and* Mehd, P. V., Estimation of Sulphides in Lime Liquors, 325

———, *and* Parker, J. G., Estimation of Glucose in Leather, 270

Blount, B., Analysis of Cement and Cement Raw Materials, 208

Blum, W., Determination of Manganese by the Bismuthate Method, 522

Boeck, P. A., New Form of Extraction Thimble, 288

———, *and* Williamson, M. A., New Type of Inorganic Filter for Laboratory Purposes, 526

Böeseken, J., *and* Waterman, H., Biochemical Method for the Estimation of Small Quantities of Salicylic Acid in Presence of Excess of *p*-Hydroxybenzoic Acid, 316

Boggs, C. R., Direct Determination of Rubber, 574

Bolton, E. R., Notes on Shrewsbury and Knapp's Process for Estimating Cocoanut Oil, 6

———, Notes on the Determination of Butter-Fat and Cocoanut Oil in Margarine, 123

———, On the Application of Adsorption to the Detection and Separation of Certain Dyes, 343

———, The Detection of Heavy Petroleum in Paints and Vegetable Oils, 249

———, The Examination of Chinese and Japanese Wood Oil, 552

Bolton, E. R., Richmond, H. D., *and* Revis, C., On the Relation between the Kirschner and Polenske Values for Margarines Containing Cocoanut or Palm Kernel Oils, 183
Bomer, A., Solidified (Hydrogenised) Oils, 452
Bömer, A., *and* Limprich, R., Glycerides Occurring in Fats and Oils: Heptadecoic Acid and its Glyceride, 411
Bono, A., Detection of Methyl Alcohol in Ethyl Alcohol and Spirits, 567
Boshart, K., *and* Bleyer, B., Gravimetric Estimation of Beryllium, 581
Boughton, E. W., *and* Walker, P. H., Fluorescence Test for Mineral and Rosin Oils, 30
Boulez, V., Analysis of Oil of Citronella, 567
Bourion, F., Separation of Iron from Titanium, 323, 469
Bousfield, W. R., Continuous Fractional Distillation of Water, 474
————, Thermo-Regulators, 108
Bowser, L. T., Volumetric Determination of Carbonic Acid, 207
————, Volumetric Estimation of Small Quantities of Potassium, 34
Bradley, C. E., *and* Tartar, H. V., Comparative Study of Methods for Determination of Hard and Total Soft Resins in the Hop, 191
Brame, J. S., Constant-Temperature Heating Apparatus for Explosives and Experiments on the Decomposition of Nitrocelluloses, 222
Bressanin, G., Applications of the Method of Precipitating Metals as Iodides, etc., from Sulphuric Acid Solution, 519
————, Detection and Estimation of Arsenic in Organic Compounds, 66, 360
————, Detection, Separation, and Estimation of Arsenic and Antimony, 65
————, Rapid Method of Purifying Sulphuric Acid, 381
————, Separation and Estimation of Arsenic and Antimony, 206
Brieger, R., Estimation of Mercury in Oxyphenyldimercuric Acetate and Mercurisalicylic Acid, 103
Brioux, C., Estimation of Mustard Oils in Oil-Cakes and Mustard Flours, 95
Brooks, B. T., Champaca Oils, 28
————, New Philippine Essential Oils, 61
Brown, D. J., *and* Schoch, E. P., Electro-Analysis of Copper, Antimony, Bismuth, and Tin with Acidified Chloride Electrolytes, 584
———, J. A., The Estimation of Small Quantities of Essential Oils in Spices, etc., Part II., 88
Browne, F., Heat Test for Chinese Wood Oil, 410
Brubaker, H. W., Modification of the Modified Winkler Method for the Determination of Sulphates in Water, 220
Bruno, A., *and* d'Auzay, P. T., Estimation of Sulphates in Solution by the Physico-Chemical Volumetric Method, 282
Bryan, H., *and* Fletcher, C. C., Modification of the Method of Mechanical Soil Analysis, 589
Bulir, J., Composition of the Fatty Oils Obtained from the Seeds of "Illicium Verum" Hook and "Illicium Religiosum" Sieb, 495
Bunzel, H. H., Measurement of the Oxidase Content of Plant Juices, 261
Burge, W. E., Separation of Rennin and Pepsin by Means of the Electric Current, 141
Burgess, H. E., On the Application of Adsorption to the Detection and Separation of Certain Dyes, 343
————, The Constituents of Oil of Savin, 298
————, The Estimation of Small Quantities of Essential Oils in Spices, etc., Part II., 90
Burgess, L. L., *and* Kamm, O., Study of Cobaltinitrites and their Application to Analytical Chemistry, 322

Burgess, W. T., An Apparatus for Testing Water by Measurement of its Electrical Conductivity, 543
——————, The Estimation of Ammonia in Carbonated Waters, 177
Burmann, J., Chemical Valuation of Digitalis, 189
Burmeister, H., *and* Hopner, W., Analysis of Commercial Casein, 493
Burr, A., Wolff, A., *and* Berberich, F. M., Parchment Paper, 465
Burrell, G. A., New Forms of Gas Analysis Apparatus, 288
——————, Use of Mice and Birds for Detecting Carbon Monoxide after Mine Fires and Explosions, 315
——————, Hall, C., Snelling, W. O., Howell, S. P., *and* Munroe, C. E., Investigations of Explosives Used in Coal Mines, 290

Cain, J. R., Estimation of Chromium and its Separation from Vanadium Steels, 146
——————, *and* Demorest, D. J., New Method for Determination of Vanadium, 286
——————, *and* Hostetter, J. C., Rapid Method for Determination of Vanadium in Steels, Ores, etc., Based on its Quantitative Inclusion by the Phosphomolybdate Precipitate, 284
Calvet, L., Alcools. Alcool, Alcool Dénaturé, Dénaturants, *Review*, 39
Camilla, S., *and* Pertusi, C., Separation and Identification of Saccharin and Dulcin in Beverages, Foods, and Drugs, 23
Campbell, F. H., Modified Explosion Eudiometer, 38
Cappa, A., *and* Pasquero, V., Detection of Caramel in Fermented Beverages, 18
Carlinfanti, E., *and* Marzocchi, P., Estimation of Saponin and Saccharin in Oil Emulsions, 22
Carney, R. J., Use of the Reagent "Tetramethyl Base," 105
Carpiaux, E, *and* Gregoire, A., Estimation of Oxalic Acid in Vegetable Substances, 564
Cary-Curr, H. J., Extraction Apparatus, 427
Celichowski, K., Mitscherlich, E. A., *and* Fischer, H., Estimation of Small Quantities of Potassium, 214
Chadwick, G., *and* Rutherford, E., New Compensation Method for the Comparison of Quantities of Radium, and Some Applications of this Method, 473
Chapman, A. C., Estimation of Furfural by Means of Fehling's Solution, 46
——————, Standards for Malt Vinegar, 123
——————, The Estimation of Ammonia in Carbonated Waters, 177
——————, The Estimation of Small Quantities of Essential Oil in Spices, etc., Part II., 89
——————, The Examination of Chinese and Japanese Wood Oil, 543
——————, The Separation of Arsenic from Antimony and Other Metals, with Some Applications to Toxicological Work, 237
——————, *and* Siebold, A., On the Application of Adsorption to the Detection and Separation of Certain Dyes, 339
Charitschkoff, —, New Reaction for Organic Bases, 319
Chauvin, A. C., Estimation of Gum in Syrups, 94
Chenu, G., *and* Leduc, E., Chaux, Ciments, Plâtres, *Review*, 161
Chercheffsky, N., Analysis of Mixtures of Ceresin and Paraffin Wax, 59
Chesneau, G., Principes Théoriques et Pratiques d'Analyse Minérale, *Review*, 436
Christopher, H., Simple Apparatus for Sublimation in a Vacuum, 38
Chwala, A., *and* Colle, E., Analysis of Higher Oxides of Lead, 148
Clarke, L. Combustion of Volatile Organic Liquids, 361
Classen, A., Theorie und Praxis der Massanalyse, *Review*, 534
Clausmann, P., *and* Gautier, A., Detection and Estimation of Minute Quantities of Fluorine in Waters, Minerals, and Living Tissues, 517
Cockburn, J., Gardiner, A. D., *and* Black, J. W., The Gravimetric Separation of Zinc and Nickel, 443

Coert, J. H., *and* Baudisch, O., Angeli's Reaction for Aldehydes, 359
Cohen, J. B., *and* Ruston, A. G., Soot: Its Character and Composition, 63
Cohn, R., Detection of Nitric Acid in Watered Fruit Juices, 21
Cole, M. J., *and* Cross, M. I., Modern Microscopy, *Review*, 117
Colle, E., *and* Chwala, A., Analysis of Higher Oxides of Lead, 148
Collins, S. H., Combined Governor and Gauge for Regulating Flow of Gas, 330
———, Rate of Evolution of Hydrocyanic Acid from Linseed under Digestive Conditions, 313
———, S. W., The Separation of Arsenic from Antimony and Other Metals, with Some Applications to Toxicological Work, 229
Collison, R. R., Estimation of Inorganic Phosphorus in Plant Substances, 472
Colombano, A., Solanidine from "Solanum Tuberosum," 497
Connah, J., On the Application of Adsorption to the Detection and Separation of Certain Dyes, 343
———, The Composition of Milk, 302
Corelli, O., *and* Grün, A., Saponification of Fats by Sulphuric Acid, 256
Cormimbœuf, H., Estimation of Glycyrrhizic Acid in Liquorice Juice, 138
Corper, H. J., Errors in the Estimation of Cholesterol by the Ritter Method: Effect of Autolysis on Cholesterol, 199
Coste, J. H., A Drying Oven, 385
———, Shelbourn, E. J., *and* Andrews, E. R., The Examination of Mixtures Containing Petroleum, 8
Costes, G., New Method of Estimating Caffeine, 401
Cottrell, F. G., Electrically-Heated Microscope Slide, 592
Cram, M. P., Determination of Melting-Point with the Aid of the Microscope, 428
Crawford, W. G., *and* Lenher, V., New Colorimetric Method for Titanium, 590
Cribb, C. H., Notes on the Determination of Butter-Fat and Cocoanut Oil in Margarine, 123
Croad, R. B., *and* Agnew, J. W., The Constituents of Oil of Savin, 295
Cross, C. F., *and* Bevan, E. J., Estimation of Cellulose in Woods and Textile Fibres, 510
———, M. I., *and* Cole, M. J., Modern Microscopy, *Review*, 117
———, W. E., *and* Taggart, W. G., Direct Determination of Sucrose in the Presence of Reducing Sugars, 451
Crown, H. A., *and* Porst, C. E. G., Lintner's Polarimetric Method for the Determination of Starch, 493
Crozier, W. J., *and* Baskerville, C., Determination of Ozone by Means of Cadmium Potassium Iodide, 587
Cumming, A. C., Notes on Thermostats, Shaking and Stirring Apparatus, etc., 70
Curry, B. E., *and* Smith, J. O., Short Method for Determination of Soluble Arsenic in Commercial Lead Arsenates, 206

Dam, W. van, Estimation of the Activity of Commercial Rennet, 564
Davis, C., *and* Foucar, J. L., Rapid Volumetric Method for Estimation of Free Sulphur, 151
———, L., *and* Woodman, A. G., Estimation of Benzaldehyde in Maraschino Cherries and Liqueur, 446
———, —, *and* Sadler, —, Allen's Commercial Organic Analysis, *Review*, 598
Davitt, S., Studies on Soil Humus, 466
Day, F. E., *and* Baker, J. L., The Iodimetric Titration of Sulphites in Presence of Alcohol and Sugars, 439
Demorest, D. J., Bismuthate Method for Manganese, 148
———, New Method for Determination of Vanadium in Steel, 285
———, *and* Cain, J. R., New Method for Determination of Vanadium, 286

Denigès, G., Sensitive Reaction of Free Bromine, 581

Denis, W., *and* Folin, O., New Colorimetric Method for the Determination of Vanillin in Flavouring Extracts, 501

Desgrez, —, *and* Feuillié, —, Estimation of Urea, 27

Deussen, E., *and* Eger, B., Examination of Copaiba Oil, 347

Dewey, F. P., Direct Determination of Small Amounts of Platinum in Ores and Bullion, 281

——————, Sampling Gold Bullion, 517

Dey, B. B., *and* Sen, H. K., Detection of Nitrates in Presence of Nitrites, 149

Dibdin, W. J., New Hand Photometer, 429

Diedrichs, A., *and* Sprinkmeyer, H., Analytical Constants of Some Vegetable Fats, 349

——————————————, Bromine Absorption of Certain Vegetable Oils and Fats, 403

Dittrich, M., *and* Eitel, W., Improvements in the Ludwig-Sipöcz Method for the Estimation of Water in Silicates, 380

——————, *and* Leonhard, A., Estimation of Ferrous Iron in Silicate Rocks, 146

Ditz, H., *and* Bardach, F., Estimation of Phenol and *p*-Cresol in the Presence of Each Other, 62

Dodge, F. D., Estimation of Cineol in Volatile Oils, 461

Domergue, A., Analysis of Mustard Flour, 55

Donau, J., Estimation of Sulphur and of Halogens in Small Quantities of Organic Substances, 216

—————, The Quantitative Manipulation of Small Quantities of Precipitates, 68

Dormane, J., Estimation of Phosphorus in Wine, 24

Dreaper, W. P., Allen's Commercial Organic Analysis, *Review*, 385

Dubourg, E., Analyses of Wines from the Department of Gironde, 352

Duboux, M., *and* Dutoit, P., L'Analyse des Vins par Volumétrie Physico-Chimique, *Review*, 600

Duclaux, J., Mechanism of Coagulation, 353

Dumont, J., New Method for the Physical Analysis of Soils, 36

Dunbar, P. B., *and* Bacon, R. F., Apparatus for Extracting Liquids with Immiscible Solvents, and for Measurement of Evolved Gases, 69

——————————————, Determination of Malic Acid, 29

Dunoyer, L., Apparatus for Rapid Distillation of Mercury in Vacuo, 528

Dunstan, A. E., *and* Thole, F. B., A Textbook of Practical Chemistry for Technical Institutes, *Review*, 73

Dupont, J., *and* Labaune, L., Direct Estimation of Geraniol in Citronella Oil, 363

Dutoit, P., *and* Duboux, M., L'Analyse des Vins par Volumétric Physico-Chimique, *Review*, 600

Dyer, B., The Analysis of Lithopone, 241

—————, The Phosphomolybdate Estimation of Phosphoric Acid in Soils, 136

Ebler, E., Estimation of Radium in Minerals and Rocks, 323, 421

Eger, B., *and* Deussen, E., Examination of Copaiba Oil, 347

Eichhorn, W., *and* Henrich, F., Apparatus for Rapid Removal of Nitrogen from Gas Mixtures by Sparking, 223

Eitel, W., *and* Dittrich, M., Improvements in the Ludwig-Sipöcz Method for the Estimation of Water in Silicates, 380

Ekecrantz, T., *and* Erickson, S., Estimation of Urea in Urine by Riegler's Method. A Correction Factor, 357

——————, *and* Palme, H., Apparatus for Measuring Liquids in an Atmosphere Free from Carbon Dioxide, 222

Ekecrantz, T., *and* Sodermann, K. A., Modification of Riegler's Method for the Estimation of Urea in Urine, 100

Ellerton, J. G., Estimation and Elimination of Sulphur Compounds in Commercial Benzol, 100

Elliot, T. G., Volumetric Estimation of Sulphur in Iron and Steel, 67

Elsdon, G. D., *and* Evers, N., The Bacteriology of Aerated Water, 395

——————————, The Estimation of Ammonia in Carbonated Waters, 173

——————, *and* Liverseige, J. F., Tests for Linseed Oil and its Adulterants, 192

Emery, W. O., Volatile Acidity of Gum Tragacanth compared with that of Indian Gum, 318

Erickson, S., *and* Ekecrantz, T., Estimation of Urea in Urine by Riegler's Method, A Correction Factor, 357

Ericson, E. J., Technical Analysis of Spelter, 521

Escard, J., New Densivolumeter for Determination of Density of Solids, 328

Evans, J., The Composition of Milk, 302

Evers, N., The Detection and Estimation of Arachis Oil, 487, 537

————, *and* Elsdon, G. D., The Bacteriology of Aerated Water, 395

——————————, The Estimation of Ammonia in Carbonated Waters, 173

Evershed, F., An Apparatus for Testing Water by Measurement of its Electrical Conductivity, 542

————, —, On the Application of Adsorption to the Detection and Separation of Certain Dyes, 344

Eynon, L., *and* Lane, J. H., The Estimation of Furfural by Means of Fehling's Solution, 41

Falk, K. G., *and* Nelson, J. M., Experiments with the Castor-Bean Lipase, 316

——————————, Studies on Enzyme Action. II. Hydrolytic Action of Some Amino Acids and Polypeptides on Certain Esters, 353

Fallada, O., *and* Strohmer, F., Inversion of Sucrose Solutions by Means of Ammonium Chloride, 576

Fanto, R., Studies on Flour, 495

Fasal, H., Colorimetric Method for the Estimation of Tryptophane, and the Quantity of Tryptophane in Horny Tissue and Other Proteins, 565

Federow, G. von, Crystallo-Chemical Analysis: A New Method of Chemical Analysis, 526

Feinberg, B. G., Determination of Aldehydes, 568

Feldstein, L., Refractive Index of Beeswax, 348

Feuillié, —, *and* Desgrez, —, Estimation of Urea, 27

Fichtenholz, A., Application of the Biochemical Method to the Analysis of Uva-Ursi Leaves, 24

Fieber, R., Rapid and Exact Estimation of Tungsten in Ferro-Tungsten, 220

Fiehe, J., *and* Stegmuller, —, Examination of Methods used in the Analysis of Honey, 449

Fieldner, A. C., *and* Stanton, F. M., Methods of Analysing Coal and Coke, 317

Filaudeau, G., Analyses of Wines from Eastern France, 352

Fillinger, F. von, New Process for the Estimation of Sugars, 63

Finch, G., Volumetric Estimation of Sulphuric Acid, Nitric Acid, and Nitrous Acid, in Nitrating and Waste Acids, 381

Fincke, H., Formic Acid in Honey, 258

Fischer, A., *and* Weise, J., Electrolytic Estimation of Molybdenum. Part I., 420

Fischer, F., *and* Ploetze, H., Electrical Furnace for Studying the Course of Reactions under Great Pressure, 288

Fischer, H., *and* Meyer-Betz, F., Urobilinogen of Urine and Ehrlich's Reaction for Aldehydes, 28
———, *and* Mitscherlich, E. A., Potash Analysis (Potassium-Sodium Cobaltinitrite Method), 588
———, Mitscherlich, E. A., *and* Celichowski, K., Estimation of Small Quantities of Potassium, 214
———, K., *and* Greunert, O., Influence of Certain Preservatives on the Keeping Properties and Composition of Butter, 52
———, W. M., *and* Steinbach, N., New Volumetric Method for the Estimation of Nitrites, and for the Separation of Nitrous from Nitric Acid, 586
Fiske, A. H., New Silver Spiral for Use in Organic Elementary Analysis, 289
Fittipaldi, E. H., Detection of Albumoses in Urine, 24
Fitzpatrick, R. M., Estimation of Water in Soap, 31
Flanders, F. F., *and* Folin, O., New Method for the Estimation of Hippuric Acid in Urine, 356
Fletcher, C. C., *and* Bryan, H., Modification of the Method of Mechanical Soil Analysis, 589
———, J., *and* Tyrer, D., Easily Adjustable Vapour Thermostat, 430
Florentin, D., *and* Kling, A., General Method for the Estimation of Tartaric Acid, 516
Fol, J. G., Relationship between the Amount of Resins and the Viscosity of Rubber Solutions, 575
Folin, O., *and* Denis, W., New Colorimetric Method for the Determination of Vanillin in Flavouring Extracts, 501
———, *and* Flanders, F. F., New Method for the Estimation of Hippuric Acid in Urine, 356
———, *and* Macallum, B., Blue Colour-Reaction of Phosphotungstic Acid with Uric Acid and Other Substances, 355
Fortini, V., Estimation of Magnesia in Admixture with Magnesium Carbonate, 211
Foucar, J. L., *and* Davis, C., Rapid Volumetric Method for Estimation of Free Sulphur, 151
Fouchet, A., Estimation of Formic Acid in the Presence of Other Acids by Means of Potassium Permanganate, 270
Fourneau, —, *and* Piettre, —, Proximate Analysis of Complex Lipoids by Alcoholysis, 463
Frank, E., Estimation of Sugar in Blood and Urine, 356
———, G. H., *and* Perkin, A. G., Analysis of Indigos Containing Starch, 319
Frankforter, G. B., *and* Frary, F. C., New Method for Estimation of Alcohol, 565
Frary, F. C., *and* Frankforter, G. B., New Method for Estimation of Alcohol, 565
French, H. F., New Method for Determining the Melting-Point of Pitch, 62
Frerichs, K., *and* Behre, A., Fruit Juice Statistics for the Year 1911, 94
Fresenius, W., *and* Grünhut, L., Estimation of Extract in Wort and Beer, 502
Frey, H. C., *and* Smith C., E. Volumetric Method for the Determination of Phenol-*p*-Sulphonic Acid, 464
Fric, R., Alteration of Nitrocellulose and Nitrocellulose Powders by Heat, 104
Friedrichs, F., Modified Soxhlet Extraction Apparatus with Distillation Arrangement, 596
———, New Extraction Apparatus, 39
Fries, J. A., Adiabatic Device for Bomb Calorimeter, 326
Friese, W., Estimation of Benzoic Acid in Margarine, Butter, and Other Fats, 92
Fuller, H. C., Estimation of Camphor, 93
Fürth, O. von, *and* Lank, E., Physico-Chemical Method for Determining the Changes Taking Place in Meats, 455

Galecki, A. von, Zsigmondy, R., *and* Wilke-Dorfürt, E., Application of Ultrafiltration in Analytical Chemistry, 223

Gardiner, A. D., Cockburn, T., *and* Black, J. W., The Gravimetric Separation of Zinc and Nickel, 443
Garratt, F., Rapid Determination of Vanadium in Steel, 283
Garrett, F. C., Examination of Bituminous Materials, 266
Gastaldi, C., *and* Ponzio, G., Relationship between the Iodine Value and the Structure of Acids of the Oleic Series, 463
Gautier, A., *and* Clausmann, P., Detection and Estimation of Minute Quantities of Fluorine in Waters, Minerals, and Living Tissues, 517
Gayon, U., *and* Laborde, J., Vins, *Review*, 599
Gerum, J., *and* Lehmann, P., Estimation of the Total Solids of Vinegar, 260
Giolitti, F., *and* Marantonio, M., Modification of the Method of Arnold and Hardy for the Rapid Estimation of Sulphur in Siderurgical Products, 283
Golblum, H., Estimation of Perchloric Acid in Certain Perchlorates, 33
————, *and* Gunther, H., Electrolytic Estimation of Manganese and its Separation from Iron, 419
Golodetz, A., Separation of Liquids, the Boiling-Points of which lie close together, or which Form Mixtures of Minimum Boiling-Point, 272
Gortner, R. A., *and* Rost, C. O., Estimation of Total Manganese in Soils, 423
Goutal, E., *and* Mahler, P., Use of Oxygen under Pressure for Estimating Carbon in Ferro-Alloys, 379
Graber, H. T., Observations on the Assay of Digestive Enzymes, 57
Grandmougin, E., *and* Havas, E., Titration of Azo Dyestuffs by Means of Hydrosulphite, 568
Gratz, O., *and* Náray, A., Comparative Value of the Catalase, Reductase, and Leucocyte Tests in Detecting Mastitis, 401
Gray, W. H., *and* Orton, K. J. P., The Determination of Nitric and Nitrous Acids in Acetic Acid Solution. The Stability of Nitric Acid in Acetic Acid Solution, 303
Gregoire, A., Study of the Acidity of Soils, 466
————, Study on the Acidity of Soils, 422
————, *and* Carpiaux, E., Estimation of Oxalic Acid in Vegetable Substances, 564
Greunert, O., *and* Fischer, K., Influence of Certain Preservatives on the Keeping Properties and Composition of Butter, 52
Grimbert, L., *and* Morel, J., Estimation of the Acidity of Urine, 141
Grimme, C., Estimation of Fat by Extraction with Cold Solvents, 102
Grün, A., *and* Corelli, O., Saponification of Fats by Sulphuric Acid, 256
Grünhut, L., *and* Fresenius, W., Estimation of Extract in Wort and Beer, 502
Grutterink, A., Micro-Chemical Detection of Certain Alkaloids, 137
Gschwender, B., Honcamp, F., Müllner, H., *and* Reich, M., Experiments on the Value of Ordinary and Treated Sawdust for Animal Nutrition, 559
Guasco, M. A., Apparatus for the Detection of Carbon Monoxide, 426
Guerbet, M., Detection of Tertiary Alcohols, 197
Guerithault, E., Detection and Estimation of Small Quantities of Copper in Plants, 313
Gulik, H. van, Modified Butyrometer Tube for the Estimation of Fat in Cheese, 221
Gunther, A., Wine Statistics for the Year 1909-1910, 96
————, H., *and* Golblum, H., Electrolytic Estimation of Manganese and its Separation from Iron, 419
Gurney, H. P., Method of Measuring Absolute Viscosity, 154
Gury, E., Estimation of Crude Fibre, 447
Gwiggner, A., Estimation of Tar in Producer Gas, 275

Hadley, H. F., *and* McFarland, D. C., Use of Higher Phenols in Testing for Free Lime in Portland Cement, 582

Haldane, J. S., Methods of Air Analysis, *Review*, 335
Hall, C., *and* Howell, S. P., Investigations of Fuse and Miners' Squibs, 291
———, Snelling, W. O., Howell, S. P., Burrell, G. A., *and* Munroe, C. E., Investigations of Explosives Used in Coal Mines, 290
Halphen, G., Huiles et Graisses Végétales Comestibles, *Review*, 292
Hamalainen, J., Detection of Savin Oil in Toxicological Cases, 357
Hampshire, C. H., Volumetric Analysis for Students of Pharmaceutical and General Chemistry, *Review*, 390
Hanslian, R., *and* Abderhalden, E., Ester Method for the Detection of Mono-Amino Acids together with Polypeptides, 272
Harned, H. S., *and* Hildebrand, J. H., Rapid Determination of Magnesia in Limestone by Means of the Hydrogen Electrode, 585
Harrison, E. F., *and* Self, P. A. W., Estimation of Nicotine in Tobacco, 311
Hart, —, *and* Tottingham, —, Acid-Soluble Phosphorus Compounds of Some Feeding Materials, 193
Hartel, F., *and* Kirschner, A., Analysis of Candied Lemon Peel, 20
Hartwich, C., *and* Wichmann, A., Apparatus for the Estimation of Adulterants in Vegetable Powders, and Notes on Starch Granules, 529
Hasenbaümer, J., König, J., *and* Hassler, C., Estimation of Colloids in Arable Soils, 98
Haslam, R. T., Detection of Small Quantities of Alkali Bicarbonate in Presence of Much Normal Carbonate, 372
Hasselbalch, K. A., Electrometric Determination of the Reaction of Liquids Containing Carbon Dioxide, 416
Hassler, C., König, J., *and* Hasenbaümer, J., Estimation of Colloids in Arable Soils, 98
Havas, E., *and* Grandmougin, E., Titration of Azo Dyestuffs by Means of Hydrosulphite, 568
Hawk, P. B., Modification of Wohlgemuth's Method for the Quantitative Investigation of the Pancreas Function, 315
Hawkins, E. M., On the Relation between the Kirschner and Polenske Values for Margarines Containing Cocoanut or Palm Kernel Oils, 188
————, The Estimation of Ammonia in Carbonated Waters, 177
Hawley, H., *and* Arnaud, F. W. F., Notes on the Determination of Butter Fat and Cocoanut Oil in Margarine, 122
Heath, G. L., Estimation of Oxygen and Occluded Gases in Copper, 375
Heaven, G. S., *and* Wilson, L. P., New Oxygen Absorption Method for Oils, 412
Hébert, A., Characteristics of Palm Oil from Different Varieties of Palm, 55
Heczko, A., Estimation of Sulphur in Pyrites, 34
Hehner, O., The Examination of Chinese and Japanese Wood Oil, 552
Heide, C. von der, *and* Schwenk, E., Critical Remarks on the Direct Estimation of the Total Solids of Wine, 311
————, ———— Estimation of Succinic and Malic Acids in Wine, 453
————, *and* Schwenk, J., Estimation of Phosphoric Acid in Wines, 453
Hempel, W., Estimation of Hydrogen and Methane in Gas Mixtures, 518
Henderson, J. B., Freezing-Point of Milk, 307
Hendrixson, W. S., Perchloric Acid in Electrochemical Analysis, 279
Henrich, F., *and* Eichhorn, W., Apparatus for Rapid Removal of Nitrogen from Gas Mixtures by Sparking, 224
Hensel, M., Estimation of Phenol in Urine, 356
Herbig, W., Assay of Sodium Peroxide for Bleaching Purposes, 476
Herzberg, W., "Pergamyn" and Imitation Parchment Papers, 144
Herzfeld, E., Estimation of Small Quantities of Bilirubin, 352
————, Estimation of Sugar in Blood, 263
Hesse, A., *and* Kooper, W. D., Nature of Peroxydase, 504

Hetper, J., Action of Permanganate on Organic Substances, 366
Heublein, O., *and* Tillmans, J., Estimation of Free Carbon Dioxide in Water by Titration with Alkalis in the Presence of Phenolphthaleïn, 582
Heubner, W., *and* Rosenberg, H., Photographic Estimation of the Intensity Distribution in Blood Spectra, 313
Hewitt, J. T., Dyes and Colouring Matters, Dyestuffs of Groups 6 to 12, Colouring Matters of Natural Origin and Analysis of Colouring Materials, *Review*, 387
——, *and* Mann, G. R., The Estimation of Ferric Iron in Presence of Certain Organic Substances, 179
Hibbert, H., Method for Determining the Relative Reactivity of Organic Compounds, 204
——, Quantitative Estimation of Hydroxy-, Amino-, and Imino-Derivatives of Organic Compounds by Means of the Grignard Reagent, and the Nature of the Changes Taking Place in Solution, 201
——, *and* Wise, A., New Method for the Separation of Tertiary from Secondary and Primary Amines, 201
Hildebrand, J. H., *and* Harned, H. S., Rapid Determination of Magnesia in Limestone by Means of the Hydrogen Electrode, 585
Hill, C. A., The Estimation of Small Quantities of Essential Oils in Spices, etc. Part II., 90
Hinds, J. I. D., Precipitation of the Copper Arsenic Group and Separation of its Divisions, 376
——, Volatility of Arsenious Chloride, 580
Hinks, E., A Flour Improver, 90
Hinrichsen, F. W., *and* Kindscher, E., Hübener's Bromide Method for the Estimation of Caoutchouc, 202
Hoagland, R., Determination of Gliadin or Alcohol-Soluble Protein in Wheat-Flour, 19
Hoffmann, J. F., Contrast Flow Aspirator, 481
Hofmann, K. A., *and* Storm, D., Tetraformal Trisazine from Formaldehyde and Hydrazine Hydrate, a New Reducing Agent for Analytical Use, 369
Hofwimmer, F., Valuation of Dynamite Glycerol, 103
Honcamp, F., Gschwender, B., Müllner, H., *and* Reich, M., Experiments on the Value of Ordinary and Treated Sawdust for Animal Nutrition, 559
Hopfner, W., *and* Burmeister, H., Analysis of Commercial Casein, 493
Hoseason, J. H., *and* Klug, O., Preliminary Notes on the Examination of Various Stick-Lacs and Shellacs, 204
Hostetter, J. C., *and* Cain, J. R., Rapid Method for Determination of Vanadium in Steels, Ores, etc., Based on its Quantitative Inclusion by the Phosphomolybdate Precipitate, 284
Houston, A. C., Metropolitan Water Board, Sixth Annual Report, 597
——, Seventh Report on Research Work of the Metropolitan Water Board, 1912, 195
——, The Softening, Purification, and Sterilisation of Water Supplies (Metropolitan Water Board, Eighth Report on Research Work), 334
Howell, S. P., *and* Hall, C., Investigations of Fuse and Miner's Squibs, 291
——, Hall, C. Snelling, W. O., Burrell, G. A., *and* Munroe, C. E., Investigations of Explosives Used in Coal Mines, 290
Hryntschak, T., Method for the Estimation of Hippuric Acid in Urine, 507
Hübener, G., Hübener Bromide Method for the Estimation of Caoutchouc, 509
——, Hübener Bromide Method for the Estimation of Rubber, 465
Hübner, J., Bleaching and Dyeing of Vegetable Fibrous Materials, *Review*, 535
Huggenberg, C., *and* Stadlinger, H., "Sapometer," 479

Hühn, F., *and* König, J., Estimation of Cellulose in Woods and Textile Fibres, 268, 510

Huish, H. C., *and* Richmond, H. D., The Souring of Milk, 168

Huizinga, A., Estimation of Nitrates and Nitrites in Water by Schlœsing's Method, 212

Huntly, G. N., An Apparatus for Testing Water by Measurement of its Electrical Conductivity, 542

Hutchinson, H. B., Valve for Filter Pumps, 480

Hyde, A. L., Separation of Nitroglycerine from Nitro-Compounds, 572

Ingle, H., Notes on the Gum Resins, 270

Ishida, M., *and* Neuberg, C., Estimation of Sugars in Natural Products, 145

Issoglio, G., *and* Possetto, G., Estimation of Saccharin in Foods, 194

Jacobs, W. A., *and* Allen, J. C., Determination of Water in Petroleum and its Products, 573

————————, Physical and Chemical Properties of the Petroleums of the San Joaquin Valley of California, 104

Jacobson, C. A., Delicate Method for Determining Minute Quantities of Chlorophyll, 503

————, *and* Marchlewski, L., Methods for Determining Neo- and Allo-Chlorophyll in the Presence of One Another, 460

Jacomet, L., "Matières Tannants Cuirs," *Review*, 40

Jadin, F., *and* Astruc, A., Amounts of Arsenic in Vegetable Foods, 254

Jago, W., *and* Jago, W. C., The Technology of Bread-Making: Chemistry and Analysis of Materials Used in Bread-Making and Confectionery, *Review*, 115

Jahn, F., Estimation of Small quantities of Iron in Organic Substances, 32

James, C., Separation of the Rare Earths, 380

———, *and* Smith, T. O., Quantitative Separation of Lanthanum from Yttrium, 469

–, T. V., New Method for the Separation of Thorium, 219

––, *and* Whittemore, C. F., Quantitative Determination of Yttrium, 382

Jamieson, G. S., Estimation of Hydrazine, 364

Jannasch, P., Use of a Mixture of Nitric Acid and Hydrogen Peroxide in Analytical Work, 212

————, *and* Routala, O., Quantitative Separation of Copper from Arsenic, Aluminium, Zinc, Tungsten, and Tin by Means of Hydrogen Peroxide in Presence of Sodium Hydroxide and Sugar, 210

Jansen, F. C. M., *and* Reicher, L. T., Refractometric Estimation of the Strength of Formaldehyde Solutions, 202

Järvinen, K. K., Estimation of Sulphuric Acid, 152

Jeanneret, B., *and* Wunder, M., Separation of Zirconium from Iron and Aluminium, and a New Method for the Analysis of Ferro-Zircon, 36

Jenkins, J. H. B., An Apparatus for Testing Water by Measurement of its Electrical Conductivity, 543

—————, The Analysis of Lithopone, 241

—————, The Examination of Chinese and Japanese Wood Oil, 552

Jessen-Hansen, H., Influence of the Concentration of Hydrogen Ions on the Baking Qualities of Flour, 257

Johnson, A. E., The Analyst's Laboratory Companion, Fourth Edition, *Review*, 437

Jolles, A., Behaviour of Invert Sugar in the Presence of Alkali and Hydrogen Peroxide, 28

———, Detection of Glycuronic Acid in Diabetic Urine, 507

———, Detection of Lævulose in the Presence of Dextrose, 507

Jolles, A., Estimation of Cane-Sugar in Foods, etc., 493
———, Estimation of Cane-Sugar in Urine in the Presence of Other Sugars, 506
———, Physico-Chemical Basis of the Seliwanoff Lævulose Reaction, 367
———, Sensitive Test for the Detection of Albumin in Urine, 505
Jona, T., Detection of Small Quantities of Chloral in Presence of Chloroform, 254
———, *and* Rimini, E., Estimation of Formaldehyde, 269
Jones, C. H., Activity of Organic Nitrogen as Measured by the Alkaline Permanganate Method, 365
———, H. C., The Freezing-Point, Boiling-Point, and Conductivity Methods, *Review*, 598
Jong, A. W. K. de, Estimation of Cinnamic Acid by Means of Bromine in Aqueous Solution, 102

Kafka, E., Potassium Iodide and Mercurous Nitrate as Sensitive Reagents for Tungsten and Molybdenum, 378
Kalusky, L., Estimation of Husk in Cocoa Powders, 402
Kamm, O., *and* Burgess, L. L., Study of Cobaltinitrites and their Application to Analytical Chemistry, 322
Karaoglanow, L., Gravimetric Estimation of Magnesium, 471
———, Volumetric Estimation of Manganese, 471
Kasanki, A., Separation of Peroxydase from Catalase, 315
Kautzsch, K., *and* Abderhalden, E., Esterification of Monamino Acids by Means of Ethyl Iodide—Separation of Pyrrolidonecarboxylic Acid from Glutamic Acid, 360
Keane, C. A., *and* Austin, W. L., The Analysis of Lithopone, 238
Kedrovitsch, D. D., Crude Phytosterol of Cocoanut Oil and the Crude Cholesterol of Butter Fat, and the Detection of Cocoanut Oil in Butter Fat, 497
Kelley, W. P., *and* McGeorge, W., Determination of Humus in Hawaiian Soil, 513
Kendall, A., Determination of Copper: A Modification of the Iodine Method, 66
———, E. C., Determination of Iodine in the Presence of Other Halogens and Organic Matter, 418
———, New Method for the Determination of the Reducing Sugars, 205
Kennedy, C., Modification of the Sweeney Method for Crude Fibre, 447
Kerb, J., *and* Neuberg, C., Precipitant for Amino-Acids, 359
Kichline, F. O., Determination of Carbon in Steel by Direct Combustion in the Newest Form of Shimar Crucible, with the Aid of a Perforated Clay Disc, 521
Kindscher, E., *and* Hinrichsen, F. W., Hübener's Bromide Method for the Estimation of Caoutchouc, 202
Kirschner, A., *and* Hartel, F., Analysis of Candied Lemon Peel, 20
Kittel, J., *and* Wogring, A., Estimation of Boric Acid in Nickel Baths, 277
Klason, P., Method for the Estimation of Selenium in Sulphur and Pyrites, 67
———, *and* Mellquist, H., Iodimetric Estimation of Selenium in Sulphur and Pyrites, 215
Klimont, J., Composition of Hare Fat, 255
Kling, A., *and* Florentin, D., General Method for the Estimation of Tartaric Acid, 516
Klug, O., *and* Hoseason, J. H., Preliminary Notes on the Examination of Various Stick-Lacs and Shellacs, 204
Knapp, A. W., *and* Shrewsbury, H. S., Notes on Shrewsbury and Knapp's Process for Estimating Cocoanut Oil, 3
Kneczynski, J., Analysis of High-Grade Tungsten Alloys, 424
Knecht, E., *and* Batey, J. P., Modification of the Beckmann Apparatus, 384
Kober, P. A., *and* Sugiura, K., Iodimetric Estimation of Copper, 375
Koch, E., Lendrich, K., *and* Schwarz, L., Hydnocarpus Fat, 20
Kochmann, U., *and* Strecker, W., Gasometric Estimation of Ether and Chloroform Vapours in Air, 513

Koenig, W., Estimation of Methyl Alcohol in Admixture with Alcohol, and Especially in Spirits, 514
Kohn, E., Examination of Meal, 139
Kohn-Abrest, E., Process of Extracting Alkaloids from Syrups, 138
Kojo, K., Composition of Hens' Eggs, 25
Kolb, A., Volumetric Estimation of Chloric Acid and Chlorates, 373
König, J., Structural Elements of Cellular Membranes, 503
———, *and* Hühn, F., Estimation of Cellulose in Woods and Textile Fibres, 268, 510
———, Hasenbäumer, J., *and* Hassler, C., Estimation of Colloids in Arable Soils, 98
Kooper, W. D., Are the Alkalinity and Peroxydase of Milk Identical ? 141
———, *and* Hesse, A., Nature of Peroxydase, 504
Köpke, O., Occurrence of Arsenic in Gelatin, 94
Koss, M., New Reagent for Thorium, 382
Kreis, H., Detection and Estimation of Formic Acid in Syrups, 558
Krueger, F., *and* Moeller, M., Absorption of Ultraviolet Rays in Ozone, and its Application to the Estimation of Ozone in Small Concentrations, 472
Kuhn, A., Beckmann Thermometer with a Modified Arrangement for Setting the Mercury Thread, 429
Kullgren, C., Diphenylamine Test for Gun-Cotton, 512
———, Estimation of Sulphur in Nitrocellulose, 152, 273
Kurtenacker, A., Elementary Analysis, 508

Labaune, L., *and* Dupont, J., Direct Estimation of Geraniol in Citronella Oil, 363
Laborde, J., *and* Gayon, U., Vins, *Review*, 599
Lake, H. B., "Luminator" Treatment of Water, 154
Lamb, A. B., *and* Marden, J. W., Quantitative Determination of Perchlorates, 374
Landau, M., Application of Light Energy to the Study of Certain Problems in Analytical Chemistry, 478
Lane, J. H., *and* Eynon, L., The Estimation of Furfural by Means of Fehling's Solution, 41
Lank, E., *and* von Fürth, O., Physico-Chemical Method for Determining the Changes Taking Place in Meats, 455
Lasègue, G., Estimation of Chlorous Acid (Chlorites), 583
Leduc, E., *and* Chenu, G., Chaux, Ciments, Plâtres, *Review*, 161
Lehmann, F., *and* Müller, A., Estimation of Cinnamein in Peru Balsam, 93
———, *and* Rupp, E., Estimation of Arsenic, 415
———, P., *and* Gerum, J., Estimation of the Total Solids in Vinegar, 260
———, *and* Schowalter, E., Polarimetric Estimation of Starch in Sausages, 498
Lendrich, K., *and* Nottbohm, F. E., Composition of Honey from Various Countries, 53
———, Koch, E., *and* Schwarz, L., Hydnocarpus Fat, 20
Lenher, V., *and* Crawford, W. G., New Colorimetric Method for Titanium, 590
———, *and* Meloche, C. C., Influence of Lead on the Ferrocyanide Titration of Zinc, 585
Lenk, E., *and* Mondschein, G., Simultaneous Effect of Alcohol and a Neutral Salt on the Sensitiveness of Phenolphthaleïn, 321
Leonhard, A., *and* Dittrich, M., Estimation of Ferrous Iron in Silicate Rocks, 146
Lessing, R., New Apparatus for the Coking Test of Coal, 383
———, The Souring of Milk, 172
Levene, P. A., *and* Van Slyke, D. D., Composition and Properties of Glycin Picrate, and Separation of Glycin from Alanin, 570
Levy, A. G., The Estimation of Carbon in Steel, 392

Levy, L. A., Rapid Estimation of Carbon Monoxide, 106
Leys, A., Estimation of Hydrocarbons in Beeswax and Carnauba Wax, 348
Limprich, R., *and* Bömer, A., Glycerides Occurring in Fats and Oils: Heptadecoic Acid and its Glyceride, 411
Lindner, P., A Substitute for the Petri Dish for Cultivating Fungi and for Biological Analysis, 561
Ling, A. R., Estimation of Furfural by Means of Fehling's Solution, 46
————, The Estimation of Small Quantities of Essential Oils in Spices, etc., Part II., 90
Lintner, C. J., Polarimetric Estimation of Starch, 351
Lipman, C. B., Effect of Ignition on the Solubility of Soil Phosphates, 523
Litterscheid, F., Use of Arsenious Acid in Volumetric Analysis. II., Estimation of Mercury, 321
Little, H. F. V., The Estimation of Manganese by the Bismuthate Method, 554
Liverseege, J. F., *and* Elsdon, G. D., Tests for Linseed Oil and its Adulterants, 192
Lott, F. E., *and* Matthews, C. G., The Diastatic Power of Malt, 58
Lowe, W. F., Estimation of Dirt in Milk, 450
Lücker, F., *and* Neubauer, H., Estimation of Phosphoric Acid by Von Lorenz's Method, 149
Lunge, G., Sulphuric Acid and Alkali, *Review*, 337

Macallum, B., *and* Folin, O., Blue Colour-Reaction of Phosphotungstic Acid with Uric Acid and Other Substances, 355
Macara, T., Notes on Shrewsbury and Knapp's Process for Estimating Cocoanut Oil, 6
————, The Analysis of Lithopone, 241
————, The Separation of Arsenic from Antimony and Other Metals, with Some Applications to Toxicological Work, 237
Macfadden, A. W. J., On the Work of Inspectors of Foods, 71
MacNider, G. M., Method for Determining the Value of Commercial Starches for Use in Cotton Mills, 368
Madinaveitia, A., *and* Willstätter, R., Estimation of Glycerol in Fats, 571
Mahler, P., *and* Goutal, E., Use of Oxygen under Pressure for Estimation of Carbon in Ferro-Alloys, 379
Mäkinen, E., Estimation of Alkalis in Silicates by Fusion with Calcium Chloride, 146
Malvezin, P., Estimation of Tannin in Wines, 195
Mann, G. R., *and* Hewitt, J. T., The Estimation of Ferric Iron in Presence of Certain Organic Substances, 179
Marantonio, M., and Giolitti, F., Modification of the Method of Arnold and Hardy for the Rapid Estimation of Sulphur in Siderurgical Products, 283
Marc, R., Estimation of the Concentration of Colloidal Solutions with the New Interferometer for Liquids, 382
Marchlewski, L., *and* Jacobson, C. A., Methods for Determining Neo- and Allo-Chlorophyl in the Presence of One Another, 460
Marcille, R., Tests for Determining the Purity of Oil of Turpentine, 371
Marcusson, J., Differentiation between Natural and Petroleum-Residue Asphalts, 407
————, Estimation of Petroleum Spirit and Benzene Hydrocarbons in Turpentine Oil, 274
Marden, J. W., *and* Lamb, A. B., Quantitative Determination of Perchlorates, 374
Marriage, E., Detection of Adulteration by Colloidal Chemical Methods, 557
Martin, N. H., Iodine Content of Thyroid Glands, 456
Marzocchi, P., *and* Carlinfanti, E., Estimation of Saponin and Saccharin in Oil Emulsions, 22

Masson, I., *and* Ramsay, W., Analysis of the Waters of the Thermal Springs of Bath, 478
Matthews, C. G., *and* Lott, F. E., The Diastatic Power of Malt, 58
McBain, J. W., Use of Phenolphthaleïn as an Indicator, 320
McBride, R. S., Standardisation of Potassium Permanganate Solution by Sodium Oxalate, 280
McCay, Le R. W., Action of Boiling Sulphuric Acid on Platinum, 590
McFarland, D. C., *and* Hadley, H. F., Use of Higher Phenols in Testing for Free Lime in Portland Cement, 582
McGeorge, W., *and* Kelley, W. P., Determination of Humus in Hawaiian Soil, 513
McIlhiney, P. C., Method of Examining Chinese Wood Oil, 410
McNamara, W., *and* Bannister, C. O., On the Effect of Lime on the Ammonium Molybdate Method of Lead Assay, 242
McNeil, H. C., Calcium Carbide Method for Estimation of Moisture, 475
Mehd, P. V., *and* Blockley, J. R., Estimation of Sulphides in Lime Liquors, 325
Meissner, W., Comparative Examination of Engler's, Redwood's, and Saybolt's Viscosimeters, 223
Méker, P., Soude-Potasse Sels, *Review*, 75
Mélikoff, P., Separation of Phosphomolybdates from Silicomolybdates, 107
————, *and* Becaia, M., Estimation of Phosphoric Acid in Presence of Colloidal Silicic Acid, 214
Mellquist, H., *and* Klason, P., Iodimetric Estimation of Selenium in Sulphur and Pyrites, 215
Meloche, C. C., *and* Lenher, V., Influence of Lead on the Ferrocyanide Titration of Zinc, 585
Menzies, A. W. C., *and* Potter, P. D., Titration of Arsenic Acid, 580
Merwin, H. E., Determination of the Density of Minerals by Means of Rohrbach Solution of Standard Refractive Index, 148
Meyer, H., *and* Polstorff, K., Detection and Estimation of Halides in the Presence of Cyanides by Means of Formaldehyde, 461
Meyer-Betz, F., *and* Fischer, H., Urobilinogen of Urine and Ehrlich's Reaction for Aldehydes, 28
Meyerfeld, J., Occurrence of Methylcyclopentenolon in Pyroligneous Acid, 319
Michel, F., Detection of Blood in Urine and Other Physiological Fluids, 506
Milford, L. R., Determination of Lithium, 470
Miller, E. H., A New Method for the Detection and Estimation of Small Quantities of Nitrous Acid, 345
————, Composition of Australian Sweetened Condensed Milk, 49
————, Composition of Australian (Victorian) Milk, 47
————, The Aldehyde Figure of Butter, 50
Milliau, E., Detection of Carbon Bisulphide in Oils, 101
Mitscherlich, E. A., *and* Fischer, H., Potash Analysis (Potassium Sodium Cobaltinitrite Method), 588
————, Celichowski, K., *and* Fischer, H., Estimation of Small Quantities of Potassium, 214
Moeller, M., *and* Krueger, F., Absorption of Ultraviolet Rays in Ozone, and its Application to the Estimation of Ozone in Small Concentrations, 472
Mohr, O., Use of the Zeiss Interferometer in the Technical Analysis of Flue Gases, 427
Mondschein, G., *and* Lenk, E., Simultaneous Effect of Alcohol and a Neutral Salt on the Sensitiveness of Phenolphthaleïn, 321
————, J., Estimation of Lactic Acid and β-Hydroxybutyric Acid in Mixtures of the Same, 409
————, Estimation of Lactic Acid in the Presence of Proteins, 403

Monier-Williams, G., Note on a New Preservative for Milk, Cream, etc., 178

——————, Report to the Local Government Board on Analyses and Methods of Detection of Certain Proprietary Substances Sold as Preservatives for Milk, Cream, etc., 155

——————, G. W., Report to the Local Government Board on the Nature of the Colouring Matter of Flour and its Relation to Processes of Natural and Artificial Bleaching, 596

Moormann, A., *and* Bleyer, B., Volumetric Estimation of Beryllium, 276

Morel, J., *and* Grimbert, L., Estimation of the Acidity of Urine, 141

Morey, G. W., Benzoic Acid as an Acidimetric Standard, 458

——————, New Apparatus for Vacuum Sublimation, 290

Morres, W., Relations Between the Reductase Test, Acidity, and Alcohol Test in Milk, 26

——————, Tests for Ascertaining the Freshness of Milk, 25

Moser, L., Titration of Copper Salts with Titanous Chloride, 516

——————, *and* Perjatel, F., Estimation of Arsenious Acid with Permanganate in Presence of Hydrochloric Acid, 417

——————, Separation of Arsenic from Antimony and Other Metals by Means of Methyl Alcohol in a Current of Air, 414

Müller, A., *and* Lehmann, F., Estimation of Cinnamein in Peru Balsam, 93

Müllner, H., Honcamp, F., Gschwender, B., *and* Reich, M., Experiments on the Value of Ordinary and Treated Sawdust for Animal Nutrition, 559

Munroe, C. E., Hall, C., Snelling, W. O., Howell, S. P., *and* Burrell, G. A., Investigations of Explosives Used in Coal Mines, 290

Murmann, E., Simplification of Gravimetric Analysis, 31

Náray, A., *and* Gratz, O., Comparative Value of the Catalase, Reductase, and Leucocyte Tests in Detecting Mastitis, 401

Nash, L. M., On the Application of Adsorption to the Detection and Separation of Certain Dyes, 344

——————, The Composition of Milk, 301

——————, The Detection and Estimation of Arachis Oil, 537

-, The Examination of Chinese and Japanese Wood Oil, 553

Neave, G. B., Solubilities of the Lead Salts of the Higher Fatty Acids in Ether and in Petroleum Ether, 399

Nelson, J. M., *and* Falk, K. G., Experiments with the Castor-Bean Lipase, 316

——————, Studies on Enzyme Action. II. Hydrolytic Action of Some Amino-Acids and Polypeptides on Certain Esters, 353

Neubauer, H., *and* Lücker, F., Estimation of Phosphoric Acid by Von Lorenz's Method, 149

Neuberg, C., *and* Ishida, M., Estimation of Sugars in Natural Products, 145

——————, *and* Kerb, J., Precipitant for Amino-Acids, 359

——————, *and* Schewket, O., Detection of Combined Glycuric Acid in Normal Urine, 562

——————, Polarimetric Estimation of Glucosamine in Ovomucoid and Pseudomucin, 561

Neumann, D., Estimation of Bitter Principles in Wort and Beer, 92

——————, D., Bitter Resins of Hops in Worts and Beers and their Volumetric Estimation, 139

Niermeyer, J., Estimation of Sulphur in Illuminating Gas, 152

Nitchie, C. C., Rapid Method for Determination of Sulphur in Roasted Blende, 150

Nockmann, E., Detection of Adulterants in Saffron, 309

Normann, W., Estimation of the Acetyl Value, 508

Nottbohm, F. E., *and* Lendrich, K., Composition of Honey from Various Countries, 53
———, *and* Weisswange, W., Estimation of Iron in Milk, 308
Nowak, H., Estimation of Sucrose in Condensed Milk, 450
Noyes, A. A., System of Qualitative Analysis for the Common Elements: Part V., Acidic Constituents, 322

Odell, A. F., Oil of the Southern Cypress, 364
Oppler, B., Estimation of Dextrose in Urine and Blood, 140
Orton, K. J. P., *and* Gray, W. H., The Determination of Nitric and Nitrous Acids in Acetic Acid Solution. The Stability of Nitric Acid in Acetic Acid Solution, 303
Otto, J., Estimation of Oxalic Acid in the Leaves of Conifers, 204

Palitzsch, S., Application of Methyl-Red to the Colorimetric Estimation of Hydrogen Ion Concentrations, 142
———, Measurement of Concentration of Hydrogen Ions in Sea Water, 425
Palme, H., *and* Ekecrantz, T., Apparatus for Measuring Liquids in an Atmosphere Free from Carbon Dioxide, 222
Palmer, C., Geochemical Interpretation of Water Analysis, 107
———, R. M., *and* Allen, W. S., Determination of Small Quantities of Arsenic by the Gutzeit Test, 579
Parisot, J., *and* Vernier, —, Toxicity of Mushrooms: Their Hæmolytic Action, 563
Parker, J. G., *and* Blockley, J. R., Estimation of Glucose in Leather, 270
Parkes, A. E., Estimation of Furfural by Means of Fehling's Solution, 46
———, The Composition of Milk, 301
Parr, S. W., New Calorimeter Bomb, with Special Advantages as to Material of Construction and Method of Operation, 592
Parry, E. J., Food and Drugs. Vol. I.: The Analysis of Food and Drugs, *Review*, 156
———, ———. Vol. II.: The Sale of Food and Drugs Acts, 1875-1907, *Review*, 158
———, The Estimation of Small Quantities of Essential Oils in Spices, etc., Part II., 89
Pasquero, V., *and* Cappa, A., Detection of Caramel in Fermented Beverages, 18
———, *and* Zuco, F. M., Clavicepsin: A New Glucoside in Ergot of Rye, 19
Paterson, J. H., Modification of the Landsberger Boiling-Point Apparatus, 326
Patrick, W. A., *and* Walker, W. H., Determination of Oxygen in Iron and Steel by Reduction in an Electric Vacuum Furnace, 586
———, W. W., *and* Wilsnack, G. C., Volumetric Determination of Tin, 477
Patten, A. J., *and* Robinson, C. S., Neutral Ammonium Citrate Solution, 372
Pawlenko, A., Elm-Seed Fat, 201
Pénau, H., Estimation of Essential Oil in Preparations of Mustard, 451
Pence, C. M., Bromine and Iodine Methods for the Volumetric Estimation of Cresol, 408
Perjatel, F., *and* Moser, L., Estimation of Arsenious Acid with Permanganate in Presence of Hydrochloric Acid, 417
———, Separation of Arsenic from Antimony and Other Metals by means of Methyl Alcohol in a Current of Air, 414
Perkin, A. G., *and* Frank, G. H., Analysis of Indigos Containing Starch, 319
Persoz, J., *and* Beltzer, J. G., Les Matières Cellulosiques, *Review*, 74
Pertusi, C., *and* Camilla, S., Separation and Identification of Saccharin and Dulcin in Beverages, Foods, and Drugs, 23
Peters, A. W., A Critical Study of Sugar Analysis by Copper Reduction Methods, 413

Peters, A. W., Sources of Error and the Electrolytic Standardisation of the Conditions of the Iodide Method of Copper Analysis, 277
Pfyl, B., *and* Turnau, R., Preparation of Milk Serums and their Use in the Analysis of Milk, 450
Philip, R., Investigations on Mercury Fulminate, 318
Philippe, E., New Sublimation Apparatus and its Application in Food Analysis, 428
Pickering, S. U., Copper Fungicides, 262
Piest, C., Characteristics of Cotton Wax, 202
———, Detection of Pine Oil in Turpentine Oil, 143
Pieszcgek, E., Device for Preventing the Bumping of Boiling Liquids, 153
Piettre, —, *and* Fourneau, —, Proximate Analysis of Complex Lipoids by Alcoholysis, 463
Ploetze, H., *and* Fischer, F., Electrical Furnace for Studying the Course of Reactions under Great Pressure, 288
Polenske, E., Detection of Cocoanut Oil in Butter and Lard, 93
Pollard, W. B., The Detection of Heavy Petroleum in Paints and Vegetable Oils, 247
Polstorff, K., *and* Meyer, H., Detection and Estimation of Halides in the Presence of Cyanides by Means of Formaldehyde, 461
Ponzio, G., *and* Gastaldi, C., Relationship between the Iodine Value and the Structure of Acids of the Oleic Series, 463
Porst, C. E. G., *and* Crown, H. A., Lintner's Polarimetric Method for the Determination of Starch, 498
Porter, C. W., Determination of Molecular Weights of Volatile Liquids, 595
Posetto, G., *and* Issoglio, G., Estimation of Saccharin in Foods, 194
Potter, P. D., *and* Menzies, A. W. C., Titration of Arsenic Acid, 580
Potts, H. E., The Chemistry of the Rubber Industry, *Review*, 225
Power, F. B., *and* Rogerson, H., Composition of Scammony Root and of Scammony Resin, 194
Pratt, D. S., Estimation of Citric Acid, 199
———, W. R., Determination of Iron in (Pharmaceutical) Scale Preparations, 306
Pribram, B. O., Quantitative Estimation of *l*-β-Hydroxybutyric Acid in Urine and Blood, 314
Price, J. M., Method for Separating Mixtures of the Seven Coal-Tar Dyestuffs Allowed (U.S.A.) to be Employed for Colouring Foodstuffs, 19
Prost, E., *and* Ubaghs, M., Influence of Metallic Carbonates on the Estimation of the Volatile Substances in Coal, 360
Putt, E. B., Micro-Chemical Tests for the Identification of Some of the Alkaloids, 400

Ramsay, C. F., New Method for Determining the Tryptic Value of Pancreatin, 26
———, W., *and* Masson, I., Analysis of the Waters of the Thermal Springs of Bath, 478
Ransome, A. O., Effect of Free Fatty Acid on Specific Gravity of Some Fatty Oils, 410
Raquet, D., Estimation of Essential Oil in Mustard, 309
Rây, P. R., *and* Sen, H. K., Reaction of Hydrazine and Hydroxylamine with Ferricyanides, and New Methods for Estimating Hydrazine and Ferricyanides, 417
Redman, I. V., *and* Rhodes, E. O., Rapid Methods for Determining Phenol, 515
Reed, E. E., Estimation of Halogens in Organic Compounds, Including a Device for the Combustion of Volatile Substances, 461
Reich, M., Honcamp, F., Gschwender, B., *and* Müllner, H., Experiments on the Value of Ordinary and Treated Sawdust for Animal Nutrition, 559
Reicher, L. T., *and* Jansen, F. C. M., Refractometric Estimation of the Strength of Formaldehyde Solutions, 202

Reid, E. E., Analysis of Organic Substances by an Electrical Method, 331
Revis, C., Note on the Detection of Benzoic Acid in Milk, 346
———, Notes on Shrewsbury and Knapp's Process for Estimating Cocoanut Oil, 6
———, On the Relation between the Kirschner and Polenske Values for Margarines Containing Cocoanut or Palm Kernel Oils, 183
———, The Separation of Arsenic from Antimony and Other Metals, with Some Applications to Toxicological Work, 237
———, The Composition of Milk, 301
———, The Souring of Milk, 171
Rhodes, E. O., *and* Redman, I. V., Rapid Methods for Determining Phenol, 515
———, F. H., Detection and Determination of Cyanogen and Hydrogen Cyanide, 511
Rice, E. W., Expanded Meissl-Hiller Table for Invert Sugar, 575
Richards, P. A. E., Notes on Shrewsbury and Knapp's Process for Estimating Cocoanut Oil, 7
———, ———, The Detection and Estimation of Arachis Oil, 537
———, T. W., *and* Shipley, J. W., New Method for Quantitative Analysis of Solutions by Precise Thermometry, 333
Richmond, H. D., On the Application of Adsorption to the Detection and Separation of Certain Dyes, 344
———, The Composition of Milk, 298
———, The Detection and Estimation of Arachis Oil, 537
———, The Effect of Lime on the Ammonium Molybdate Method of Lead Assay, 247
———, *and* Huish, H. C., The Souring of Milk, 168
———, Bolton, E. R., *and* Revis, C., On the Relation between the Kirschner and Polenske Values for Margarines Containing Cocoanut or Palm Kernel Oils, 183
Richter, E., Determination of Bleaching Quality of Sulphite Pulp, 511
———, O., Estimation of Fat in Cocoa by Means of Zeiss' Refractometer, 495
———, R., Estimation of Acetaldehyde in Paraldehyde, 265
Rideal, —, A Method for Determining the Amount of Insoluble Particles in Raw Rubber, 15
Riegler, E., Colorimetric Method for the Estimation of Uric Acid, 358
Rimini, E., *and* Jona, T., Estimation of Formaldehyde, 269
Rindell, A., Solubility Estimations in Agricultural Chemistry, 37
Ringelmann, M., Sterilisation of Water and Dairy Materials by Ultra-Violet Rays, 196
Rippetoe, J. R., Determination of the Digestive Value of Papain, 405
Rivett, A. C. D., Estimation of Water in Butter, 51
Roberts, C. C., Determination of Water in Molasses, 560
Robertson, T. B., Refractive Indices of Solutions of Certain Proteins. VI. The Proteins of Ox Serum : A New Optical Method for the Estimation of the Various Proteins in Ox Serum, 355
Robin, L., Detection of Minute Traces of Boric Acid by Means of Tincture of Mimosa Flowers, 558
Robinson, C. S., *and* Patten, A. J., Neutral Ammonium Citrate Solution, 372
Rogerson, H., *and* Power, F. B., Composition of Scammony Root and of Scammony Resin, 194
Rohmann, F., *and* Shmamine, T., Compounds of Ferric Salts with Albumoses, 408
Romijn, G., Estimation of the Ferrous Ion by Means of Iodine, 32
Rosenberg, H., *and* Heubner, W., Photographic Estimation of the Intensity Distribution in Blood Spectra, 313
Rosenblatt, M., Estimation of Dextrose in the Presence of Other Optically Active Substances by Bertrand's Method, 512

Rosenheim, A., Quantitative Estimation of Thorium, especially in Monazite Sand, by Means of Sodium Hypophosphate, 423
Rosenthaler, L., Colour Reaction of Alcohols and Alcoholic Hydroxyl Groups, 407
——————, The Mutarotation of Honey, 95
Ross, B. B., Electrolytic Apparatus for Use in Invert Sugar Determinations, 576
——, R., An Apparatus for Testing Water by Measurement of its Electrical Conductivity, 542
————, On the Application of Adsorption to the Detection and Separation of Certain Dyes, 343
————, The Composition of Milk, 301
————, The Constituents of Oil of Savin, 298
————, The Detection and Estimation of Arachis Oil, 537
————, The Detection of Heavy Petroleum in Paints and Vegetable Oils, 249
————, The Effect of Lime on the Ammonium Molybdate Method of Lead Assay, 247
————, The Examination of Chinese and Japanese Wood Oil, 553
——, W. H., *and* Benner, R. C., Filtration with Alundum Plates, 109
Rossi, G., Catalytic Action of Light on the Oxidation of Phenolphthalin and Phenolphthaleïn, 30
Rost, C. O., *and* Gortner, R. A., Estimation of Total Manganese in Soils, 423
Rothenfusser, S., Detection of Cane-Sugar, 493
——————, Estimation of Glycerol in Wine, 260
Routala, O., Analysis of Hydrocarbon Mixtures, 462
————, *and* Jannasch, P., Quantitative Separation of Copper from Arsenic, Aluminium, Zinc, Tungsten, and Tin by Means of Hydrogen Peroxide in Presence of Sodium Hydroxide and Sugar, 210
Rühle, J., Detection of Saponin, 350
Rupp, E., Detection of Fluorides, 32
Ruston, A. G., *and* Cohen, J. B., Soot: Its Character and Composition, 63
Rutherford, E., *and* Chadwick, G., New Compensation Method for the Comparison of Quantities of Radium, and Some Applications of this Method, 473

Sadler, —, *and* Davis, —, Allen's Commercial Organic Analysis, *Review*, 598
Salkowski, E., Behaviour of Milk when Treated with Ammonium Sulphate, and a New Method for the Estimation of Lactose, 350
—————, Trommer's Test for Sugar in Urine, 406
Salway, A. H., Alkaloidal Assay of Calabar Beans, 142
—————, Chemical Examination of Calabar Beans, 60
Sanarens, J., Composition of Guadaloupe Rum, 97
Sanders, J. M., Estimation of Sulphur in Petroleum, 217
Savage, W. G., Milk and the Public Health, *Review*, 160
Saville, W. B., The Detection and Estimation of Arachis Oil, 537
Savtori, A., Detection of Fluorine in Foods, etc., 191
Schaeffer, J. A., Colorimetric Determination of Iron in Lead and its Oxides, 520
Schapiro, A., *and* Wunder, M., Fusion of Certain Rare Earths with Alkali Carbonates, Separation of Tungsten from Iron, Beryllium, and Aluminium, 591
Schär, E., Chemical Detection of Blood in Blood-Stains, 454
Schewket, O., *and* Neuberg, C., Detection of Combined Glycuronic Acid in Normal Urine, 562
——————————, Polarimetric Estimation of Glucosamine in Ovomucoid and Pseudomucin, 561
Schidrowitz, P., A Method for Determining the Amount of Insoluble Particles in Raw Rubber, 15

Schidrowitz, P., Estimation of Furfural by Means of Fehling's Solution, 46

Schilling, H., Estimation of Chromium in Bronzes Containing Tin and Antimony, 374

Schippers, J. C., Method of Preparing Lecithin Emulsions and the Subsequent Estimation of their Strength, 307

Schirm, E., Apparatus for Controlling the Evaporation or Distillation of Liquids, and for Cutting off the Supply of Gas to a Burner after any Desired Length of Time, 221

Schirmer, W., Methods for the Estimation of Iodides, 519

Schlesinger, M. D., *and* Sherman, H. C., Studies of Amylases. IV.: A Further Investigation of the Properties of Pancreatic Amylase, 454

Schmidt, E., Analysis of Textile Finishes, 200

Schmitz, W., Estimation of Antimony in Red Rubber Goods, 64, 65

Schneider, E. C., Insoluble Carbohydrates of the Apple, 492

Schoch, E. P., *and* Brown, D. J., Electro-Analysis of Copper, Antimony, Bismuth, and Tin with Acidified Chloride Electrolytes, 584

Schott, F., Colorimetric Estimation of Salicylic Acid and Copper, 104

Schowalter, E., *and* Lehmann, P., Polarimetric Estimation of Starch in Sausages, 498

Schulz, N., Copper-Reducing Power of Normal Urine, 264

Schwarz, F., "Acid Resins" from Petroleum Residues, 515

——————, Estimation of Asphalt in Mineral Oils, Pitch, etc., 58

————, L., Lendrich, K., *and* Koch, E., Hydnocarpus Fat, 20

Schwenk, E., *and* Heide, C. von der, Critical Remarks on the Direct Estimation of the Total Solids of Wine, 311

————————————————, Estimation of Succinic and Malic Acids in Wine, 453

————————————————, Estimation of Phosphoric Acid in Wines, 453

Seidell, A., Estimation of Thymol, Salicylates, and Allied Compounds, 369

Self, P. A. W., Source of Error in the Kjedahl-Gunning Method, 203

——————, *and* Harrison, E. F., Estimation of Nicotine in Tobacco, 311

Sen, H. K., *and* Dey, B. B., Detection of Nitrates in Presence of Nitrites, 149

——————, *and* Rây, P. R., Reaction of Hydrazine and Hydroxylamine with Ferricyanides, and New Methods for Estimating Hydrazine and Ferricyanides, 417

Šetlík, M. B., *and* Žofka, J., Analysis and Testing of Rubberised Fabrics, 575

Shannon, F. L., Detection of Formic Acid in Fruit Products, 403

Shelbourn, E. T., Coste, J H., *and* Andrews, E. R., The Examination of Mixtures Containing Petroleum, 8

Sherman, H. C., Methods of Organic Analysis, *Review*, 482

————————, *and* Schlesinger, M. D., Studies of Amylases. IV. A Further Investigation of the Properties of Pancreatic Amylase, 454

Shimer, E. B., *and* Shimer, P. W., Volumetric Determination of Titanium, 590

————, P. W., *and* Shimer, E. B., Volumetric Determination of Titanium, 590

Shipley, J. W., *and* Richards, T. W., New Method for Quantitative Analysis of Solutions by Precise Thermometry, 333

Shmamine, T., *and* Rohmann, F., Compounds of Ferric Salts with Albumoses, 408

Shrewsbury, H. S., A Method of Estimating Calcium Carbonate in Soils, 128

————————, Note on a Counterfeit Gold Coin, 7

————————, Poisonous Gases from Oilfields, 486

————————, *and* Knapp, A. W., Notes on Shrewsbury and Knapp's Process for Estimating Cocoanut Oil, 3

Siebold, A., *and* Chapman, A. C., On the Application of Adsorption to the Detection and Separation of Certain Dyes, 339

Siemssen, J. A., Reaction of Gold Salts with *m*-Phenylenediamine, 469

Sievers, E., Detection of Aniline Dyes and Turmeric in Mustard, 557

Sigmond, A. von., *and* Vuk, M., Detection of Adulteration of Cayenne Pepper, 259

Sigmond, A. von., *and* Vuk, M., Composition of Capsicum, (Cayenne Pepper), 56
Silvester, H., Estimation of Nitrates in Sewage Effluents, 149
Simmonds, C., Note on the Determination of Small Quantities of Methyl Alcohol, 16
Simonis, H., *and* Thies, F. H., Estimation of Carbon by Moist Combustion, 459
Sinkinson, E., Apparatus for Automatically Decanting and Washing Precipitates, 479
Sinnatt, F. S., A Convenient Apparatus for Obtaining an Average Sample of Gas, and for Regulating the Flow of a Gas into an Evacuated Vessel, 250
———, The Use of Methylene Blue as an Indicator in Iodimetric Titrations, 252
Skinder, W., Automatic Pressure Pipette, 153
Slade, W. C., New Method for Testing Paint Films and Preservative Coatings for Iron and Steel, 213
Slawik, P., Dimethylglyoxime as a Reagent for Ferrous Salts, 102
———, Rapid Estimation of Manganese in Ferro-Tungsten, 107
Slyke, D. D. van, Conditions for the Complete Hydrolysis of Proteins, 573
———, *and* Levene, P. A., Composition and Properties of Glycin Picrate and Separation of Glycin from Alanin, 570
Smirnow, H., *and* Weinberg, B., Comparison of Methods for Determining the Viscosity of Pitch, 413
Smith, A. R., Measurement of Relative Tryptic Activity, 457
———, C. E., *and* Frey, H. C., Volumetric Method for the Determination of Phenol-*p*-Sulphonic Acid, 464
———, T. O., *and* Curry, B. E., Short Method for Determination of Soluble Arsenic in Commercial Lead Arsenates, 206
———, *and* James, C., Quantitative Separation of Lanthanum from Yttrium, 469
———, T. V., *and* James, C., New Method for the Separation of Thorium, 219
Snelling, W. O., Hall, C., Howell, S. P., Burrell, G. A., *and* Munroe, C. E., Investigations of Explosives Used in Coal-Mines, 290
Sodermann, K. A., *and* Ekecranyz, T., Modification of Riegler's Method for the Estimation of Urea in Urine, 100
Sommer, F., Application of the Formolite Reaction to the Analysis of Paraffin Wax, 274
Sonntag, P., Microscopic Differentiation of Hemp and Flax Fibres, 195
Sorbini, F., *and* Tarugi, N., Arsenic Xanthogenate and its Use in Analysis, 569
Sormani, C., Detection of Saponin in Beverages and Foods by Hæmolysis, 350
Spät, W., Method of Testing Water. Determination of the Power of Bacteria to Decompose Proteins, 264
Splittgerber, A., *and* Tillmans, J., Estimation of Potassium Nitrate in Meats, 140
Sprinkmeyer, H., *and* Diedrichs, A., Analytical Constants of Some Vegetable Fats, 349
———, Bromine Absorption of Certain Vegetable Oils and Fats, 403
Staddon, D. R., Detection of Traces of Arsenic and Antimony, 581
Stadlinger, H., *and* Huggenberg, C., "Sapometer," 479
Stanton, F. M., *and* Fieldner, A. C., Methods of Analysing Coal and Coke, 317
Steenbock, H., Estimation of Benzoic Acid, Hippuric Acid, and Phenaceturic Acid in Urine, 356
Stegmuller, —, *and* Fiehe, J., Examination of the Methods Used in the Analysis of Honey, 449
Steinbach, N., *and* Fischer, W. M., New Volumetric Method for the Estimation of Nitrites, and for the Separation of Nitrous Acid from Nitric Acid, 586
Stevens, H. P., The Analysis of Lithopone, 241
———, *and* Beadle, C., A Method for Determining the Amount of Insoluble Particles in Raw Rubber, 13

Stevenson, E. F., Zsigmondy, R., *and* Bachmann, W., Apparatus for Determination of Vapour Pressure Isothermals of Silicic Acid Gels, 328
Stiasny, E., Detection and Differentiation of Vegetable Tannins, 577
———, *and* Wilkinson, C. D., Qualitative Reactions of Vegetable Tannins, 31
Stocks, H. B., Water Analysis for Sanitary and Technical Purposes, *Review*, 482
Stokes, G. A., Note on a New Preservative for Milk, Cream, etc., 178
Storm, C. P., Effect of the Nitrotoluenes on the Determination of Nitroglycerine by Means of the Nitrometer, 572
———, D., *and* Hofmann, K. A., Tetraformal-trisazine from Formaldehyde and Hydrazine Hydrate, a New Reducing Agent for Analytical Use, 369
Stoward, F., Amyloclastic Secretory Capacities of the Embryo and Aleurone Layer of Hordeum, 97
Straub, A., Detection of Small Quantities of Zinc in Wine, 195
Strecker, W., *and* Kochmann, M., Gasometric Estimation of Ether and Chloroform Vapours in Air, 513
Strohmer, F., *and* Fallada, O., Inversion of Sucrose Solutions by Means of Ammonium Chloride, 576
Strzyzoroski, C., Occurrence of Mercury in the Hair of Persons who have Received Subcutaneous Doses of Mercury Compounds. Detection of Minute Quantities of Mercury, 562
Sugiura, K., *and* Kober, P. A., Iodimetric Estimation of Copper, 375
Sutterheim, G. A., Gravimetric and Volumetric Methods for the Analysis of Mercury-Ammonium Chloride, 33
Swanson, —, *and* Willard, —, Baking Qualities of Flour, 190

Taggart, W. G., *and* Cross, W. E., Direct Determination of Sucrose in the Presence of Reducing Sugars, 451
Takahashi, D., Estimation of Sugar in Blood, 141
Tamura, M., Loss of Fat during the Drying of Animal Tissues, 355
Tartar, H. V., *and* Bradley, C. E., Comparative Study of Methods for Determination of Hard and Total Soft Resins in the Hop, 191
Tarugi, N., *and* Sorbini, F., Arsenic Xanthogenate and its Use in Analysis, 569
Thies, F. H., *and* Simonis, H., Estimation of Carbon by Moist Combustion, 459
Thole, F. B., *and* Dunstan, A. E., A Textbook of Practical Chemistry for Technical Institutes, *Review*, 73
Thomas, P., Colour Reaction of Ammonia, 468
Thöni, J., Application of the Quantitative Precipitin Reaction in Honey Analysis, II., 404
Thuringer, V., *and* Wunder, M., Action of Dimethyl-Glyoxime on Platinum Salts, 524
———, W, Separation of Nickel and Palladium by Means of Dimethyl-Glyoxime, 379
Tibbles, W., Foods: Their Origin, Composition, and Manufacture, *Review*, 388
Tillmans, J., *and* Heublein, O., Estimation of Free Carbon Dioxide in Water by Titration with Alkalis in the Presence of Phenolphthaleïn, 582
———, *and* Splittgerber, A., Estimation of Potassium Nitrate in Meats, 140
Tillotson, E. W., Jun., Relation of the Refractive Index of Soda Lime Glasses to their Chemical Composition, 278
Tóth, J., Methods of Estimating Nicotine in Tobacco Extracts, 452
Tottingham, —, *and* Hart, —, Acid-Soluble Phosphorus Compounds of Some Feeding Materials, 193
Trapp, H., Qualitative Analysis without Hydrogen Sulphide, 377
Trautmann, W., *and* Weiss, L., Analysis of Ferro-Zircon, 220
Treadwell, W. D., Electro-Analytical Estimation of Copper in Pyrites, 468
Trenkner, —, Estimation of Gold, Silver, and Platinum, 281

Tswett, M., Detection of Carotin, 199
Turnau, R., *and* Pfyl, B., Preparation of Milk Serums and their Use in the Analysis of Milk, 450
Turrentine, J. W., Determination of Iodides by Direct Titration, 378
Tyrer, D., Latent Heats of Vaporisation of Mixed Liquids, Part III.: Mixtures of Associated with Non-Associated Liquids, and the Detection of Solvates in Mixtures of Liquids, 362
———, *and* Fletcher, J., Easily Adjustable Vapour Thermostat, 430

Ubaghs, M., *and* Prost, E., Influence of Metallic Carbonates on the Estimation of the Volatile Substances in Coal, 360
Ulrich, C., Estimation of Cocoa-Husks in Cocoa, 52
Utz, —, Detection of Other Hydrocarbons in Turpentine, 311
———, Estimation of Caoutchouc as Tetrabromide, 361
———, Notes on Serger's Reaction for Vegetable Oils, 412
———, Reaction for Detecting Sesame Oil in Other Oils, 62

Vallery, L., The Coagulation of Albumin by Heat and its Precipitation by Mercuric-Potassium Iodide. Gravimetric and Volumetric Estimations, 458
Van Dam, W., Estimation of the Activity of Commercial Rennet, 564
Vandevelde, A. J. J., Fractional Saponification of Fats, 558
Van Gulik, H., Modified Butyrometer Tube for Estimation of Fat in Cheese, 221
Van Slyke, D. D., Conditions for the Complete Hydrolysis of Proteins, 573
———, *and* Levene, P. A., Composition and Properties of Glycin Picrate and Separation of Glycin from Alanin, 570
Vaubel, W., New Reaction of Nitrous Acid, 33
Vernier, —, *and* Parisot, J., Toxicity of Mushrooms: Their Hæmolytic Action, 563
Virchow, C., Estimation of Lecithin in Medicinal Tablets, 449
Vitali, D., New Uric Acid Reaction, 276
Voelcker, J. A., The Composition of Milk, 301
Voigt, K., Rapid Method of Estimating Zinc, 35, 153
Vozárik, A., Determination of Phosphorus in Food-Stuffs and Other Organic Substances by Titration with Uranyl Solutions, 258
———, Incineration of Food-Stuffs and Other Organic Substances Prior to the Determination of their Phosphorus-Content, 257
Vuk, M., *and* Von Sigmond, A., Composition of Capsicum (Cayenne Pepper), 56
———, Detection of Adulteration of Cayenne Pepper, 259

Wahl, R., Proteolytic Enzyme of Malt, 504
Walker, P. H., Unification of Reducing Sugar Methods (a Correction for Lactose), 145
———, *and* Boughton, E. W., Fluorescence Test for Mineral and Rosin Oils, 30
———, W. H., *and* Patrick, W. A., Determination of Oxygen in Iron and Steel by Reduction in an Electric Vacuum Furnace, 586
Warunis, T. S., Estimation of Arsenic in Organic Compounds, 579
———, Estimation of Sulphur in Insoluble Sulphides, 283
Watanabe, R., Kumagawa-Suto Method of Estimating Fat in Animal Substances, 354
Waterman, H., *and* Böeseken, J., Biochemical Method for the Estimation of Small Quantities of Salicylic Acid in Presence of Excess of *p*-Hydroxybenzoic Acid, 316
Weber, H. C. P., Modified Form of Stability Test, 573
———, W., *and* Beckmann, E., Ebullioscopic Determinations at Low Temperatures (−36° to −83° C.), 287
Wedekind, E., Use of Magnesia Rods instead of Platinum Wire in Analytical Work, 222

Weinberg, B., *and* Smirnow, H., Comparison of Methods for Determining the Viscosity of Pitch, 413
Weirman, S. A., New Stability Test for Nitrocellulose Powders, 572
Weise, J., *and* Fischer, A., Electrolytic Estimation of Molybdenum. Part I., 420
Weiss, H. F., Tests to Determine the Commercial Value of Wood Preservatives, 578
——, J. M., Antiseptic Effect of Creosote Oil and Other Oils used for Preserving Timber, 56
——, L., *and* Trautmann, W., Analysis of Ferro-zircon, 220
Weisswange, W., *and* Nottbohm, F. E., Estimation of Iron in Milk, 308
Wenger, P., *and* Wunder, W., Separation of Beryllium from Aluminium, 373
White, C. H., Colorimeter for Rapid Work with Widely Varying Standards, 329
——, G. F., Viscosity of Fish Oils, 143
Whittemore, C. F., *and* James, C., Quantitative Determination of Yttrium, 382
Whymper, R., Cocoa and Chocolate: Their Chemistry and Manufacture, *Review*, 226
Wichmann, A., *and* Hartwich, C., Apparatus for the Estimation of Adulterants in Vegetable Powders and Notes on Starch Granules, 529
Wilke-Dörfurt, E., Estimation of Potassium in Potassium Silicate, 588
——, Spectroscopic Process for the Estimation of Small Quantities of Rubidium in Presence of Much Potassium, 324
——, Zsigmondy, R., *and* Von Galecki, A., Application of Ultrafiltration in Analytical Chemistry, 223
Wilkinson, C. D., *and* Stiasny, E., Qualitative Reactions of Vegetable Tannins, 31
Willard, —, and Swanson, —, Baking Qualities of Flour, 190
Willcox, —, The Separation of Arsenic from Antimony and Other Metals, with Some Applications to Toxicological Work, 237
Williams, H. E., Estimation of Ferrocyanides, 362
Williamson, M. A., *and* Boeck, P. A., New Type of Inorganic Filter for Laboratory Purposes, 526
Willstätter, R., *and* Madinaveitia, A., Estimation of Glycerol in Fats, 571
Wilsnack, G. C., and Patrick, W. W., Volumetric Determination of Tin, 477
Wilson, L. P., *and* Heaven, G. S., New Oxygen Absorption Method for Oils, 412
Winton, A. L., Method for the Determination of the "Gasoline Colour Value" of Flour, 255
Wirthle, F., Detection and Estimation of Methyl Alcohol, 364
Wise, A., *and* Hibbert, H., New Method for the Separation of Tertiary from Secondary and Primary Amines, 201
——, L. E., Refractive Index of Chinese Wood Oil, 410
Wogrinz, A., *and* Kittel, J., Estimation of Boric Acid in Nickel Baths, 277
Wöhlk, A., Detection of Pyridine Bases in Ammonia and Ammonium Salts, 413
Wolff, A., Burr, A., *and* Berberich, F. M., Parchment Paper, 465
Wolff, H., Estimation of Petroleum in Turpentine, 371
——, Examination and Valuation of Turpentine, 370
——, O., Luminescence Analysis, 526
Woodman, A. G., *and* Davis, L., Estimation of Benzaldehyde in Maraschino Cherries and Liqueur, 446
Worden, E. C., Nitrocellulose Industry, *Review*, 338
Wright, A. M., Changes in the Composition of the Nitrogenous Constituents of Meat-Extracts, 21
——, Chemical Composition of Some New Zealand Meat Extracts, 193
Wunder, M., *and* Jeanneret, B., Separation of Zirconium from Iron and Aluminium, and a New Method for the Analysis of Ferro-Zircon, 36
——, *and* Schapiro, A., Fusion of Certain Rare Earths with Alkali Carbonates, Separation of Tungsten from Iron, Beryllium, and Aluminium, 591

Wunder, M., *and* Thuringer, V., Action of Dimethylglyoxime on Platinum Salts, 524
———, W., *and* Thuringer, V., Separation of Nickel and Palladium by Means of Dimethylglyoxime, 379
————, *and* Wenger, P., Separation of Beryllium from Aluminium, 373

Yoder, P. A., Marking of Porcelain Crucibles, etc., 289

Zerewitinoff, T., Quantitative Estimation of Active Hydrogen in Organic Compounds by Magnesium-methyl Iodide, 571
Zimmerman, A., Laboratory Studies of Rennin, 405
Zimmermann, H., Laboratory Centrifuge for Quantitative Separation and Estimation of Solids and Liquids, 286
Zipfel, H., The Indole Reaction, 562
Žofka, J., *and* Šetlík, M. B., Analysis and Testing of Rubberised Fabrics, 575
Zsigmondy, R., Bachmann, W., *and* Stevenson, E. F., Apparatus for Determination of Vapour Pressure Isothermals of Silicic Acid Gels, 328
————, Wilke-Dörfurt, E., *and* Von Galecki, A., Application of Ultrafiltration in Analytical Chemistry, 223
Zuco, F. M., *and* Pasquero, V., Clavicepsin: A New Glucoside in Ergot of Rye, 19

INDEX OF SUBJECTS.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS, 18, 51, 92, 137, 189, 254, 306, 347, 400, 446, 492, 557
Acetaldehyde in Paraldehyde, Estimation of, 265
Acetate, Oxyphenyldimercuric, and Mercurisalicylic Acid, Mercury in, Estimation of, 103
Acetic Acid Solution, Nitric and Nitrous Acids in, the Determination of. The Stability of Nitric Acid in Acetic Acid Solution, 303
Acetyl Value, Estimation of the, 508
Acid, Arsenic, Titration of, 580
——, Arsenious, Use of, in Volumetric Analysis, II., Estimation of Mercury, 321
——, ——, Estimation of, with Permanganate in Presence of Hydrochloric Acid 417
——, Benzoic, as an Acidimetric Standard, 458
——, ——, Hippuric Acid, and Phenaceturic Acid in Urine, Estimation of, 356
——, ——, in Foods, Detection of, 349
——, ——, in Margarine, Butter, and Other Fats, Estimation of, 92
——, ——, in Milk, the Detection of, Note on, 346
——, Boric, in Nickel Baths, Estimation of, 277
——, ——, Minute Traces of, Detection of, by Means of Tincture of Mimosa Flowers, 558
——, Carbonic, Volumetric Determination of, 207
——, Chloric, and Chlorates, Volumetric Estimation of, 373
——, Chlorous (Chlorites), Estimation of, 583
——, Cinnamic, Estimation of, by Means of Bromine in Aqueous Solution, 102
——, Citric, Estimation of, 199
——, Colloidal Silicic, Phosphoric Acid in Presence of, Estimation of, 214
——, Content of Distilled Vinegar when Stored in Wooden Casks, Alteration of, 23
——, Fatty, Free, Effect of, on Specific Gravity of Some Fatty Oils, 410
——, Formic, in Fruit Products, Detection of, 403
——, ——, in Honey, 258
——, ——, in Syrups, Detection and Estimation of, 558
——, ——, in the Presence of Other Acids, Estimation of, by Means of Potassium Permanganate, 270
——, Gels, Silicic, Vapour-Pressure Isothermals of, Apparatus for Determination of, 328
——, Glutamic, Separation of Pyrrolidonecarboxylic Acid from. Esterification of Monamino Acids by Means of Ethyl Iodide, 360
——, Glycuronic, Combined, in Normal Urine, Detection of, 562
——, ——, in Diabetic Urine, Detection of, 507
——, Glycyrrhizic, in Liquorice Juice, Estimation of, 138
——, Heptadecoic, and its Glyceride: Glycerides Occurring in Fats and Oils, 411
——, Hippuric, Benzoic Acid, and Phenaceturic Acid in Urine, Estimation of, 356
——, ——, in Urine, Estimation of, Method for, 507
——, ——, ——, the Estimation of, New Method for, 356

Acid, Hydrochloric, Estimation of Arsenious Acid with Permanganate in Presence of, 417
——, Hydrocyanic, Rate of Evolution of, from Linseed, under Digestive Conditions, 313
——, *p*-Hydroxybenzoic, Excess of, Biochemical Method for the Estimation of Small Quantities of Salicylic Acid in Presence of, 316
——, β-Hydroxybutyric, and Lactic Acid, Estimation of, in Mixtures of the Same, 409
——, *l*-β-Hydroxybutyric, in Urine and Blood, Quantitative Estimation of, 314
——, Lactic, Analysis of, 29
——, ——, and β-Hydroxybutyric Acid, Estimation of, in Mixtures of the Same, 409
——, ——, in the Presence of Proteins, Estimation of, 403
——, Malic, Determination of, 29
——, Mercurisalicylic, and Oxyphenyldimercuric Acetate, Mercury in, Estimation of, 103
——, Nitric, and Hydrogen Peroxide, Use of a Mixture of, in Analytical Work, 212
——, ——, in Watered Fruit Juices, Detection of, 21
——, ——, Sulphuric Acid, and Nitrous Acid, in Nitrating and Waste Acids, Volumetric Estimation of, 381
——, ——, the Separation of Nitrous Acid from, and the Estimation of Nitrites, New Volumetric Method for, 586
——, Nitrous, Detection and Estimation of Small Quantities of, a New Method for, 345
——, ——, New Reaction of, 33
——, ——, Nitric Acid, and Sulphuric Acid, in Nitrating and Waste Acids, Volumetric Estimation of, 381
——, ——, the Separation of, from Nitric Acid, and the Estimation of Nitrites, New Volumetric Method for, 586
——, Oxalic, in the Leaves of Conifers, Estimation of, 204
——, ——, in Vegetable Substances, Estimation of, 564
——, Perchloric, in Certain Perchlorates, Estimation of, 33
——, ——, in Electrochemical Analysis, 279
——, Phenaceturic, Benzoic Acid, and Hippuric Acid, in Urine, Estimation of, 356
——, Phenol-*p*-Sulphonic, the Determination of, Volumetric Method for, 464
——, Phosphoric, Estimation of, by Von Lorenz's Method, 149
——, ——, in Presence of Colloidal Silicic Acid, Estimation of, 214
——, ——, in Soils, the Phosphomolybdate Estimation of, 130
——, ——, in Wines, Estimation of, 453
——, Phosphotungstic, Blue Colour-Reaction of, with Uric Acid and Other Substances, 355
——, Pyroligneous, Occurrence of Methylcyclopentenolon in, 319
——, Pyrrolidonecarboxylic, Separation of, from Glutamic Acid. Esterification of Monamino Acids by Means of Ethyl Iodide, 360
"Acid-Resins" from Petroleum Residues, 515
Acid, Salicylic, and Copper, Colorimetric Estimation of, 104
——, ——, Small Quantities of, in Presence of Excess of *p*-Hydroxybenzoic Acid, Biochemical Method for the Estimation of, 316
—— -Soluble Phosphorus Compounds of Some Feeding Materials, 193
—— Solution, Sulphuric, the Method of Precipitating Metals as Iodides, etc., from, Applications of, 519
——, Sulphuric, Boiling, Action of, on Platinum, 590
——, ——, Estimation of, 152

Acid, Sulphuric, Nitric Acid, and Nitrous Acid, in Nitrating and Waste Acids, Volumetric Estimation of, 381
--, Rapid Method of Purifying, 381
--, Saponification of Fats by, 256
Tartaric, the Estimation of, General Method for, 516
Uric, and Other Substances, Blue Colour-Reaction of Phosphotungstic Acid with, 355
-, Colorimetric Method for the Estimation of, 358
-, Reaction, New, 276
Acidic Constituents, Part V. System of Qualitative Analysis for the Common Elements, 322
Acidified Chloride Electrolytes, Electro-Analysis of Copper, Antimony, Bismuth, and Tin, with, 584
Acidimetric Standard, Benzoic Acid as an, 458
Acidity of Soils, Study of the, 466
————, Study on the, 422
———— Urine, Estimation of, 141
———, the Reductase Test, and Alcohol Test, in Milk, Relations between, 26
———, Volatile, of Gum Tragacanth compared with that of Indian Gum, 318
Acids, Amino-, in Plants, Estimation of, by Means of the Formaldehyde Method, 312
——, ———, Precipitants for, 359
——, ———, Some, and Polypeptides, Hydrolytic Action of, on Certain Esters. Studies on Enzyme Action, 353
——, Fatty, Small Quantities of, Estimation of the Molecular Weight of, 203
——, Malic and Succinic, in Wine, Estimation of, 453
——, Monamino, Esterification of, by Means of Ethyl Iodide. Separation of Pyrrolidonecarboxylic Acid from Glutamic Acid, 360
——, Mono-Amino-, together with Polypeptides, Ester Method for the Detection of, 272
——, Nitric and Nitrous, in Acetic Acid Solution, the Determination of. The Stability of Nitric Acid in Acetic Acid Solution, 303
—— of the Oleic Series, the Iodine Value and the Structure of, Relationship between, 463
——, Succinic and Malic, in Wine, Estimation of, 453
——, the Higher Fatty, in Ether and in Petroleum Ether, Solubilities of the Lead Salts of, 399
Adiabatic Device for Bomb Calorimeter, 326
Adsorption, on the Application of, to the Detection and Separation of Certain Dyes, 339
Adulterants in Saffron, Detection of, 309
- in Vegetable Powders, Apparatus for the Estimation of, and Notes on Starch Granules, 529
-, Linseed Oil and its, Tests for, 192
Adulteration, Detection of, by Colloidal Chemical Methods, 557
———— of Cayenne Pepper, Detection of, 259
Aerated Water, the Bacteriology of, 395
Africa, British West, Kola Nuts from, 258
Agricultural Chemistry, Solubility Estimations in, 37
Air, Ether and Chloroform Vapours in, Gasometric Estimation of, 513
Alanin, Separation of Glycin from, and Composition and Properties of Glycin Picrate, 570
Albumin in Urine, the Detection of, Sensitive Test for, 505
———, the Coagulation of, by Heat, and its Precipitation by Mercuric-Potassium Iodide. Gravimetric and Volumetric Estimations, 458

Albumoses, Compounds of Ferric Salts with, 408
——— in Urine, Detection of, 24
Alcohol and a Neutral Salt, Simultaneous Effect of, on the Sensitiveness of Phenolphthaleïn, 321
——— and Sugars, the Iodimetric Titration of Sulphites in Presence of, 439
———, Estimation of Methyl Alcohol in Admixture with, and especially in Spirits, 514
———, Estimation of, New Method for, 565
———, Methyl, in a Current of Air, Separation of Arsenic from Antimony and Other Metals by Means of, 414
———, ———, in Admixture with Alcohol, and especially in Spirits, Estimation of, 514
———, ———, in Ethyl Alcohol, and Spirits, Detection of, 567
———, ———, Small Quantities of, Note on the Determination of, 16
the Detection and Estimation of, 364
-- Soluble Protein or Gliadin in Wheat-Flour, Determination of, 19
-- Test, the Reductase Test, and Acidity, in Milk, Relations between, 26
Alcohols and Alcoholic Hydroxyl Groups, Colour Reaction of, 407
———, Tertiary, Detection of, 197
Alcoholysis, Proximate Analysis of Complex Lipoids by, 463
Aldehyde Figure of Butter, 50
Aldehydes, Angeli's Reaction for, 359
———, Determination of, 568
———, Ehrlich's Reaction for, and Urobilinogen of Urine, 28
Aldoses and Ketoses, Method of Distinguishing between, 266
Aleurone Layer and Embryo of Hordeum, Amyloclastic Secretory Capacities of the, 97
Alkali and Hydrogen Peroxide, Behaviour of Invert Sugar in the Presence of, 28
——— Bicarbonate, Small Quantities of, Detection of, in Presence of Much Normal Carbonate, 372
——— Carbonates, Fusion of Certain Rare Earths with. Separation of Tungsten from Iron, Beryllium, and Aluminium, 591
Alkaline Permanganate Method, Activity of Organic Nitrogen as Measured by the, 365
Alkalinity and Peroxydase of Milk : are they Identical? 141
Alkalis in Silicates, Estimation of, by Fusion with Calcium Chloride, 146
———, Titration with, in the Presence of Phenolphthaleïn, Estimation of Free Carbon Dioxide in Water by, 582
Alkaloidal Assay of Calabar Beans, 142
Alkaloids, Certain, Micro-Chemical Detection of, 137
———, Extracting from Syrups, Process of, 138
———, Some of the, Micro-Chemical Tests for the Identification of, 400
Allo- and Neo-Chlorophyll in the Presence of One Another, Methods for Determining, 460
Alloys, High-Grade Tungsten, Analysis of, 424
———, Platinum, Analysis of, 420
Aluminium, Arsenic, Zinc, Tungsten, and Tin, Quantitative Separation of Copper from, by Means of Hydrogen Peroxide in Presence of Sodium Hydroxide and Sugar, 210
——— and Iron, Separation of Zirconium from, and a New Method for the Analysis of Ferro-Zircon, 36
———, Iron, and Beryllium, Separation of Tungsten from. Fusion of Certain Rare Earths with Alkali Carbonates, 591
———, Separation of Beryllium from, 373
Alundum Plates, Filtration with, 109
Amines, Tertiary, the Separation of, from Primary and Secondary Amines, New Method for, 201

Amino-Acids in Plants, Estimation of, by Means of the Formaldehyde Method, 312
————, Precipitant for, 359
————, Some, and Polypeptides, Hydrolytic Action of, on Certain Esters. Studies on Enzyme Action, 353
Amino-, Hydroxy-, and Imino-Derivatives of Organic Compounds, Quantitative Estimation of, by Means of the Grignard Reagent, and the Nature of the Changes Taking Place in Solution, 201
Ammonia and Ammonium Salts, Pyridine Bases in, Detection of, 413
Pyridine, Separation and Estimation of, 413
, Colour Reaction of, 468
in Carbonated Waters, the Estimation of, 173
Rapid Estimation of Pyridine in Gas-Water in the Presence of, 574
Ammonium Chloride, Inversion of Sucrose Solutions by Means of, 576
———— Citrate Solution, Neutral, 372
———— Molybdate Method of Lead Assay, on the Effect of Lime on the, 242
———— Salts and Ammonia, Pyridine Bases in, Detection of, 413
———— Sulphate, Behaviour of Milk when Treated with, and a New Method for the Estimation of Lactose, 350
Amyloclastic Secretory Capacities of the Embryo and Aleurone Layer of Hordeum, 97
Amylase, Pancreatic, a Further Investigation of the Properties of. Studies of Amylases, 454
Analysis, Crystallo-Chemical: a New Method of Chemical Analysis, 526
————, Electro-Chemical, Perchloric Acid in, 279
————, Elementary, 508
————, Food, New Sublimation Apparatus, and its Application in, 428
————, Gravimetric, Simplification of, 31
————, Luminescence, 526
————, Mechanical, of Arid Soils, 588
————, Organic Elementary, New Silver Spiral for Use in, 289
————, Potash (Potassium-Sodium Cobaltinitrite Method), 588
————, ————, without Hydrogen Sulphide, 377
————, Qualitative, System of, for the Common Elements. Part V.: Acidic Constituents, 322
————, Rational, of Nitrate of Soda, 523
————, Soil, Mechanical, Modification of the Method of, 589
————, Sugar, by Copper Reduction Methods, a Critical Study of, 414
————, Technical, of Spelter, 521
————, Volumetric, Arsenious Acid in, Use of. II.: Estimation of Mercury, 321
————, Water, Geochemical Interpretation of, 107
Analytical Chemistry, Certain Problems in, Application of Light Energy to the Study of, 479
————————, Study of Cobaltinitrites, and their Application to, 322
————————, Ultrafiltration in, Application of, 223
———— Constants of Some Vegetable Fats, 349
———— Work, Use of a Mixture of Nitric Acid and Hydrogen Peroxide in, 212
——, —— of Magnesia Rods instead of Platinum Wire in, 222
Angeli's Reaction for Aldehydes, 359
Aniline Dyes and Turmeric in Mustard, Detection of, 557
Animal Nutrition, the Value of Ordinary and Treated Sawdust for, Experiments on, 559
——— Substances, Fat in, Kumagawa-Suto Method of Estimating, 354
——— Tissues, the Drying of, Loss of Fat during, 355
Annual Address by the Retiring President, 81

Antimony and Arsenic, Detection, Separation, and Estimation of, 65
——————, Separation and Estimation of, 206
——————, Traces of, Detection of, 581
——— and Other Metals, Separation of Arsenic from, by Means of Methyl Alcohol in a Current of Air, 414
————————, the Separation of Arsenic from, with Some Applications to Toxicological Work, 229
——— and Tin, Bronzes containing, Estimation of Chromium in, 374
———, Copper, Bismuth, and Tin, Electro-Analysis of, with Acidified Chloride Electrolytes, 584
——— in Red Rubber Goods, Estimation of, 64, 65
Antiseptic Effect of Creosote Oil and Other Oils Used for Preserving Timber, 56
Apparatus, etc., Abstracts, 36, 68, 108, 153, 221, 286, 326, 382, 426, 478, 526, 592
———, Boiling-Point, the Landsberger, Modification of, 326
———, Constant-Temperature Heating, for Explosives, and Experiments on the Decomposition of Nitrocelluloses, 222
———, Convenient, for Obtaining an Average Sample of Gas, and for Regulating the Flow of a Gas into an Evacuated Vessel, 250
———, Electrolytic, for Use in Invert Sugar Determinations, 576
———, Extraction, 427
———, ———, New, 39
——— for Automatic Analysis of Liquids, 480
——— for Automatically Decanting and Washing Precipitates, 479
——— for Controlling the Evaporation or Distillation of Liquids, and for Cutting off the Supply of Gas to a Burner after any Desired Length of Time, 221
——— for Determination of Vapour Pressure Isothermals of Silicic Acid Gels, 328
——— for Extracting Liquids with Immiscible Solvents, and for Measurement of Evolved Gases, 69
——— for Measuring Liquids in an Atmosphere Free from Carbon Dioxide, 222
——— for Rapid Distillation of Mercury *in Vacuo*, 528
——— for Rapid Removal of Nitrogen from Gas Mixtures by Sparking, 224
——— for the Detection of Carbon Monoxide, 426
——— for Testing Water by Measurement of its Electrical Conductivity, 538
——— for the Estimation of Adulterants in Vegetable Powders, and Notes on Starch Granules, 529
———, Gas Analysis, New Forms of, 288
———, Laboratory, Two New Pieces of, 528
———, New, for the Coking Test of Coal, 383
———, ——, for Vacuum Sublimation, 290
———, —— Sublimation, and its Application in Food Analysis, 428
———, Shaking and Stirring, Thermostats, etc., Notes on, 70
———, Simple, for Sublimation in a Vacuum, 38
———, Soxhlet Extraction, Modified, with Distillation Arrangement, 596
———, the Beckmann, Modification of, 384
Apple, Insoluble Carbohydrates of the, 492
Arable Soils, Colloids in, Estimation of, 98
Arachis Oil, the Detection and Estimation of, 487, 537
Arid Soils, Mechanical Analysis of, 588
Arnold and Hardy, the Method of, for the Rapid Estimation of Sulphur in Siderurgical Products, Modification of, 283
Arsenates, Commercial Lead, Determination of Soluble Arsenic in, Short Method for, 206
Arsenic Acid, Titration of, 580

Arsenic, Aluminium, Zinc, Tungsten, and Tin, Quantitative Separation of Copper from, by Means of Hydrogen Peroxide in Presence of Sodium Hydroxide and Sugar, 210
——— and Antimony, Detection, Separation, and Estimation of, 65
———————, Separation and Estimation of, 206
———————, Traces of, Detection of, 581
———, Estimation of, 415
——— in Gelatin, Occurrence of, 94
——— in Organic Compounds, Detection and Estimation of, 66, 360
———————, Estimation of, 579
——— in Vegetable Foods, Amounts of, 254
———, Separation of, from Antimony and Other Metals by Means of Methyl Alcohol in a Current of Air, 414
———, Small Quantities of, Determination of, by the Gutzeit Test, 579
———, Soluble, in Commercial Lead Arsenates, Short Method for Determination of, 206
———, the Separation of, from Antimony and Other Metals, with Some Applications to Toxicological Work, 229
——— Xanthogenate, and its Use in Analysis, 569
Arsenious Acid, Estimation of, with Permanganate in Presence of Hydrochloric Acid, 417
———, Use of, in Volumetric Analysis. II.: Estimation of Mercury, 321
——— Chloride, Volatility of, 580
Asphalt in Mineral Oils, Pitch, etc., Estimation of, 58
Asphalts, Natural and Petroleum-Residue, Differentiation between, 407
Aspirator, Constant Flow, 481
Atmosphere Free from Carbon Dioxide, Apparatus for Measuring Liquids in, 222
Atomic Weights, International, 1912, 1
Australian Sweetened Condensed Milk, Composition of, 49
——— (Victorian) Milk, Composition of, 47
Autolysis, Effect of, on Cholesterol; Errors in the Estimation of Cholesterol by the Ritter Method, 199
Automatic Analysis of Liquids, Apparatus for, 480
——— Pressure Pipette, 153
Automatically Decanting and Washing Precipitates, Apparatus for, 479
Azo Dyestuffs, Titration of, by Means of Hydrosulphite, 568

Bacteria in Water, Direct Enumeration of, by Means of the Ultra-Microscope, Value of, 317
———, the Power of, to Decompose Proteins, Determination of. Method of Testing Water, 264
BACTERIOLOGICAL, PHYSIOLOGICAL, ETC., ABSTRACTS, 24, 56, 97, 140, 195, 262, 312, 352, 403, 454, 503, 561
Bacteriology of Aerated Water, 395
Baking Qualities of Flour, 190
———————, Influence of the Concentration of Hydrogen Ions on, 257
Balsam, Peru, Cinnamein in, Estimation of, 93
Banana Starch, Polarimetric Estimation of, 560
Bases, Organic, New Reaction for, 319
———, Pyridine, in Ammonia and Ammonium Salts, Detection of, 413
Bassia Kernels and Fats, 54
Bath, the Waters of the Thermal Springs of, Analysis of, 478
Beans, Calabar, Alkaloidal Assay of, 142
———, ———, Chemical Examination of, 60

Beans, Edible, from Hong-Kong, 494
Beckmann Apparatus, Modification of, 384
———— Thermometer, with a Modified Arrangement for Setting the Mercury Thread, 429
Beer and Wort, Bitter Principles in, Estimation of, 92
————, Extract in, Estimation of, 502
Beers and Worts, Bitter Resins of Hops in, and their Volumetric Estimation, 139
Beeswax and Carnauba Wax, Hydrocarbons in, Estimation of, 348
————, Refractive Index of, 348
Benzaldehyde in Maraschino Cherries and Liqueur, Estimation of, 446
Benzene Hydrocarbons and Petroleum Spirit in Turpentine Oil, Estimation of, 274
Benzoic Acid as an Acidimetric Standard, 458
————, Hippuric Acid, and Phenaceturic Acid in Urine, Estimation of, 356
———— in Foods, Detection of, 349
———— in Margarine, Butter, and Other Fats, Estimation of, 92
———— in Milk, the Detection of, Note on, 346
Benzol, Commercial, Sulphur Compounds in, Estimation and Elimination of, 100
(Benzoyl Peroxide) Lucidol, Influence of, on Certain Qualitative Tests for Oils, 273
Bertrand's Method, Estimation of Dextrose in the Presence of Other Optically Active Substances by, 512
Beryllium, Gravimetric Estimation of, 581
————, Iron, and Aluminium, Separation of Tungsten from. Fusion of Certain Rare Earths with Alkali Carbonates, 591
Separation of, from Aluminium, 373
--, Volumetric Estimation of, 276
Beverages and Foods, Saponin in, Detection of, by Hæmolysis, 350
————, Fermented, Caramel in, Detection of, 18
————, Foods, and Drugs, Saccharin and Dulcin in, Separation and Identification of, 23
Bicarbonate, Alkali, Small Quantities of, Detection of, in Presence of much Normal Carbonate, 372
Bilirubin, Small Quantities of, Estimation of, 352
Biochemical Method, Application of, to the Analysis of Uva-Ursi Leaves, 24
———— for the Estimation of Small Quantities of Salicylic Acid in Presence of Excess of *p*-Hydroxybenzoic Acid, 316
Biological Analysis, the Petri Dish for Cultivating Fungi and for, Substitute for, 561
Birds and Mice, Use of, for Detecting Carbon Monoxide after Mine Fires and Explosions, 315
Bismuth, Copper, Antimony, and Tin, Electro-Analysis of, with Acidified Chloride Electrolytes, 584
Bismuthate Method, Determination of Manganese by, 522
———— for Manganese, 148
————, the Estimation of Manganese by, 554
Bitter Principles in Wort and Beer, Estimation of, 92
—— Resins of Hops in Worts and Beers and their Volumetric Estimation, 139
Bituminous Materials, Examination of, 266
Bleaching, Natural and Artificial, Processes of, Report to the Local Government Board on the Nature of the Colouring Matter of Flour, and its Relation to, 596
———— Purposes, Sodium Peroxide for, Assay of, 476
————, Quality of Sulphite Pulp, Determination of, 511
Blende, Roasted, Sulphur in, Rapid Method for Determination of, 150
Blood and Urine, Dextrose in, Estimation of, 140
————, *l*-β-Hydroxybutyric Acid in, Quantitative Estimation of, 314

Blood and Urine, Quinine in, Quantitative Estimation of, 455
————————, Sugar in, Estimation of, 356
——, Fat and Lipoids in, Estimation of, and Notes on the So-Called Lipolysis, 561
—— in Blood-Stains, Chemical Detection of, 454
—— in Urine and Other Physiological Fluids, Detection of, 506
—— Spectra, the Intensity Distribution in, Photographic Estimation of, 313
—— -Stains, Blood in, Chemical Detection of, 454
——, Sugar in, Estimation of, 141, 263
Blue Colour-Reaction of Phosphotungstic Acid with Uric Acid and Other Substances, 355
Boiling Liquids, the Bumping of, Device for Preventing, 153
—— -Point Apparatus, the Landsberger, Modification of, 326
—— Sulphuric Acid, Action of, on Platinum, 590
Bomb Calorimeter, Adiabatic Device for, 326
——, New Calorimeter, with Special Advantages as to Material of Construction and Method of Operation, 592
Books, New, 294, 601
Books, Reviews of:
  A Textbook of Practical Chemistry for Technical Institutes, 73
  Alcools. Alcool, Alcool Dénaturé, Dénaturants, 39
  Allen's Commercial Organic Analysis, 385
  ————————————, Vol. VI., 598
  An Introduction to Quantitative Analysis, 337
  Bleaching and Dyeing of Vegetable Fibrous Materials, 535
  Chaux, Ciments, Plâtres, 161
  Church's Laboratory Guide, 292
  Cocoa and Chocolate: Their Chemistry and Manufacture, 226
  Dyes and Colouring Matters, Dyestuffs of Groups 6 to 12, Colouring Matters of Natural Origin and Analysis of Colouring Materials, 387
  Food and Drugs. Vol. I. The Analysis of Food and Drugs, 156
  ————————. Vol. II. The Sale of Food and Drugs Acts, 1875-1907, 158
  Foods: Their Origin, Composition, and Manufacture, 388
  Huiles et Graisses Végétales Comestibles, 292
  L'Analyse des Vins par Volumétrie Physico-Chimique, 600
  Les Matières Cellulosiques, 74
  "Matières Tannants Cuirs," 40
  Methods of Air Analysis, 335
  ———— Organic Analysis, 482
  Milk and the Public Health, 160
  Modern Microscopy, 117
  Nitrocellulose Industry, 338
  Principes Théoriques et Pratiques d'Analyse Minérale, 436
  Soude-Potasse Sels, 75
  Sulphuric Acid and Alkali, 337
  Tables Annuelles Internationales de Constantes et Données Numériques, 483
  The Analyst's Laboratory Companion. Fourth Edition, 437
  "The Chemical World," 76
  The Chemistry of the Rubber Industry, 225
  The Freezing-Point, Boiling-Point, and Conductivity Methods, 598
  The Technology of Bread-Making: Chemistry and Analysis of Materials Used in Bread-Making and Confectionery, 115
  Theorie und Praxis der Massanalyse, 534

BOOKS, REVIEWS OF—*continued*
Vins, 599
Volumetric Analysis for Students of Pharmaceutical and General Chemistry, 390
Water Analysis for Sanitary and Technical Purposes, 482
Boric Acid in Nickel Baths, Estimation of, 277
———, Minute Traces of, Detection of, by Means of Tincture of Mimosa Flowers, 558
British West Africa, Kola Nuts from, 258
Bromide Method, Hübener, for the Estimation of Caoutchouc, 509
———, Hübener's, for the Estimation of Caoutchouc, 202
———, Hübener, for the Estimation of Rubber, 465
Bromine Absorption of Certain Vegetable Oils and Fats, 403
——— and Iodine Methods for the Volumetric Estimation of Cresol, 408
———, Detection of, by Fluorescein, 145
———, Free, Sensitive Reaction of, 581
——— in Aqueous Solution, Estimation of Cinnamic Acid by Means of, 102
Bronzes Containing Tin and Antimony, Chromium in, Estimation of, 374
Bullion and Ores, Small Amounts of Platinum in, Direct Determination of, 281
———, Gold, Sampling, 517
Bumping of Boiling Liquids, Device for Preventing, 153
Bunsen Burners, Porcelain, 426
Butter and Lard, Cocoanut Oil in, Detection of, 93
——— Fat and Cocoanut Oil in Margarine, the Determination of, Notes on, 122
———, Cocoanut Oil in, the Detection of, and Crude Phytosterol of Cocoanut Oil and the Crude Cholesterol of Butter Fat, 497
———, Margarine, and Other Fats, Benzoic Acid in, Estimation of, 92
———, Shea-, and Shea-Nuts, 496
———, the Aldehyde Figure of, 50
———, the Keeping Properties and Composition of, Influence of Certain Preservatives on, 52
———, Water in, Estimation of, 51
Butyrometer Tube, Modified, for the Estimation of Fat in Cheese, 221

Cadmium Potassium Iodide, Determination of Ozone by Means of, 587
Caffeine, New Method of Estimating, 401
Cake and Oil, Tea-Seed, 500
Calabar Beans, Alkaloidal Assay of, 142
———, Chemical Examination of, 60
Calcium Carbide, Method for Estimation of Moisture, 475
——— Carbonate in Soils, a Method of Estimating, 128
——— Chloride, Estimation of Alkalis in, Silicates by Fusion with, 146
Calorimeter Bomb, Adiabatic Device for, 326
——— Bomb, New, with Special Advantages as to Material of Construction and Method of Operation, 592
Camphor, Estimation of, 93
Candied Lemon Peel, Analysis of, 20
Cane-Sugar, Detection of, 493
——— in Foods, etc., Estimation of, 493
——— in Urine, Estimation of, in the Presence of Other Sugars, 506
Caoutchouc, Estimation of, as Tetrabromide, 361
———, the Estimation of, Hübener Bromide Method for, 509
———, ———, Hübener's Bromide Method for, 202
Capsicum (Cayenne Pepper), Composition of, 56

Caramel in Fermented Beverages, Detection of, 18
Carbohydrates, Insoluble, of the Apple, 492
Carbon Bisulphide in Oils, Detection of, 101
——— Dioxide, an Atmosphere Free from, Measuring Liquids in, Apparatus for, 222
——————, Free, in Water, Estimation of, by Titration with Alkalis in the Presence of Phenolphthaleïn, 582
——————, the Reaction of Liquids Containing, Electrometric Determination of, 416
———, Estimation of, by Moist Combustion, 459
——— in Ferro-Alloys, Use of Oxygen under Pressure for Estimating, 379
——— in Steel, Determination of, by Direct Combustion in the Newest Form of Shimer Crucible, with the Aid of a Perforated Clay Disc, 521
——————, the Estimation of, 392
——— Monoxide after Mine Fires and Explosions, Use of Birds and Mice for Detecting, 315
——————, Rapid Estimation of, 106
Carbonate, Magnesium, Magnesia in Admixture with, Estimation of, 211
————, Much Normal, Detection of Small Quantities of Alkali Bicarbonate in Presence of, 372
Carbonated Waters, Ammonia in, the Estimation of, 173
Carbonates, Alkali, Fusion of Certain Rare Earths with, Separation of Tungsten from Iron, Beryllium, and Aluminium, 591
————, Metallic, Influence of, on the Estimation of the Volatile Substances in Coal, 360
Carbonic Acid, Volumetric Determination of, 207
Carnauba Wax and Beeswax, Hydrocarbons in, Estimation of, 348
Carotin, Detection of, 199
Casein, Commercial, Analysis of, 493
Castor-Bean Lipase, Experiments with, 316
Catalase, Reductase, and Leucocyte Tests, Comparative Value of, in Detecting Mastitis, 401
———, Separation of Peroxydase from, 315
Catalytic Action of Light on the Oxidation of Phenolphthalin to Phenolphthaleïn, 30
Cathode, Mercury, Use of, particularly in the Electro-Analytical Separation of Metals, 279
Cayenne Pepper, Adulteration of, Detection of, 259
(Cayenne Pepper) Capsicum, Composition of, 56
Cellular Membranes, Structural Elements of, 503
Cellulose in Woods and Textile Fibres, Estimation of, 268, 510
Cement and Cement Raw Materials, Analysis of, 208
———, Portland, Free Lime in, Use of Higher Phenols in Testing for, 582
——— Raw Materials and Cement, Analysis of, 208
Centrifuge, Laboratory, for Quantitative Separation and Estimation of Solids and Liquids, 286
Ceresin and Paraffin Wax, Mixtures of, Analysis of, 59
Ceylon Tobaccos, Analyses of, 500
Champaca Oils, 28
Changes taking place in Meats, Physico-Chemical Method for Determining, 455
Charcoal as a Clarifying Agent in the Estimation of Sugar in Urine, Use of, 57
Cheese, the Estimation of Fat in, Modified Butyrometer Tube for, 221
Chemical Analysis, a New Method of: Crystallo-Chemical Analysis, 526
Chemistry, Agricultural, Solubility Estimations in, 37
Cherries and Liqueur, Maraschino, Benzaldehyde in, Estimation of, 446

Chinese and Japanese Wood Oil, the Examination of, 543

——— Wood Oil, Heat Test for, 410

——————, Method of Examining, 410

——————, Refractive Index of, 410

Chloral, Small Quantities of, Detection of, in Presence of Chloroform, 254

Chlorates, and Chloric Acid, Volumetric Estimation of, 373

Chloride, Ammonium, Inversion of Sucrose Solutions by Means of, 576

———, Arsenious, Volatility of, 580

———, Calcium, Estimation of Alkalis in Silicates by Fusion with, 146

———, Electrolytes, Acidified, Electro-Analysis of Copper, Antimony, Bismuth, and Tin with, 584

———, Mercury-Ammonium, the Analysis of, Gravimetric and Volumetric Methods for, 33

———, Titanous, Titration of Copper Salts with, 516

(Chlorites), Chlorous Acid, Estimation of, 583

Chloroform, Small Quantities of Chloral in Presence of, Detection of, 254

———— Vapours and Ether in Air, Gasometric Estimation of, 513

Chlorophyll, Minute Quantities of, Delicate Method for Determining, 503

Chlorous Acid (Chlorites), Estimation of, 583

Cholesterol, Effect of Autolysis on; Errors in the Estimation of Cholesterol by the Ritter Method, 199

————, the Crude, of Butter Fat, and Crude Phylosterol of Cocoanut Oil, and the Detection of Cocoanut Oil in Butter Fat, 497

Chromium, Estimation of, and its Separation from Vanadium Steels, 146

———— in Bronzes Containing Tin and Antimony, Estimation of, 374

Cineol in Volatile Oils, Estimation of, 461

Cinnamein in Peru Balsam, Estimation of, 93

Cinnamic Acid, Estimation of, by Means of Bromine in Aqueous Solution, 102

Citric Acid, Estimation of, 199

Citronella Oil, Geraniol in, Direct Estimation of, 363

————, Oil of, Analysis of, 567

Clarifying Agent in the Estimation of Sugar in Urine, Use of Charcoal as, 57

Clavicepsin: A New Glucoside in Ergot of Rye, 19

Coagulation, Mechanism of, 353

———— of Albumin by Heat and its Precipitation by Mercuric-Potassium Iodide. Gravimetric and Volumetric Estimations, 458

Coal and Coke, Methods of Analysing, 317

—— -Mines, Explosives Used in, Investigations of, 290

—— -Tar Dyestuffs, the Seven, Allowed (U.S.A.) to be Employed for Colouring Foodstuffs, Mixtures of, Method for Separating, 19

——, the Coking Test of, New Apparatus for, 383

——, the Volatile Substances in, the Estimation of, Influence of Metallic Carbonates on, 360

(Cobaltinitrite Method, Potassium Sodium), Potash Analysis, 588

Cobaltinitrites, and their Application to Analytical Chemistry, Study of, 322

Cocoa, Cocoa-Husks in, Estimation of, 52

———, Fat in, Estimation of, by Means of Zeiss' Refractometer, 495

——— -Husks in Cocoa, Estimation of, 52

——— Powders, Husk in, Estimation of, 402

Cocoanut Oil and Butter Fat in Margarine, the Determination of, Notes on, 122

———— in Butter and Lard, Detection of, 93

———— ———— Fat, the Detection of, and Crude Phytosterol of Cocoanut Oil and the Crude Cholesterol of Butter Fat, 497

————, Shrewsbury and Knapp's Process for Estimating, Notes on, 3

Cocoanut Oil or Palm Kernel Oils, Margarines Containing, on the Relation between Kirschner and Polenske Values for, 183
Coin, a Counterfeit Gold, Note on, 7
Coke and Coal, Methods of Analysing, 317
Coking Test of Coal, New Apparatus for, 383
Cold Solvents, Estimation of Fat by Extraction with, 102
Colloidal Chemical Methods, Detection of Adulteration by, 557
———— Silicic Acid, Phosphoric Acid in Presence of, Estimation of, 214
———— Solutions, the Concentration of, Estimation of, with the New Interferometer for Liquids, 382
Colloids in Arable Soils, Estimation of, 98
Colorimeter for Rapid Work with Widely Varying Standards, 329
Colorimetric Determination of Iron in Lead, and its Oxides, 520
———— Estimation of Hydrogen Ion Concentrations, Application of Methyl-Red to, 142
———— ———— Phenols in Effluents, 30
Salicylic Acid and Copper, 104
———— Method for the Estimation of Tryptophane, and the Quantity of Tryptophane in Horny Tissue and Other Proteins, 565
———— Method for the Estimation of Uric Acid, 358
———— ————, New, for the Determination of Vanillin in Flavouring Extracts, 501
for Titanium, 590
Colour Reaction, Blue, of Phosphotungstic Acid with Uric Acid and Other Substances, 355
———— of Alcohols and Alcoholic Hydroxyl Groups, 407
———— of Ammonia, 468
Colouring Matter of Flour, the Nature of, and its Relation to Processes of Natural and Artificial Bleaching, Report to the Local Government Board on, 596
Combustion of Volatile Organic Liquids, 361
Commercial Benzol, Sulphur Compounds in, Estimation and Elimination of, 100
———— Casein, Analysis of, 493
———— Lead Arsenates, Determination of Soluble Arsenic in, Short Method for, 206
———— Rennet, the Activity of, Estimation of, 564
———— Starches for Use in Cotton Mills, Method for Determining the Value of, 368
———— Value of Wood Preservatives, Tests to Determine, 578
Compensation Method, New, for the Comparison of Quantities of Radium, and Some Applications of this Method, 473
Complex Lipoids, Proximate Analysis of, by Alcoholysis, 463
Condensed Milk, Sucrose in, Estimation of, 450
————, Sweetened, Australian, Composition of, 49
Conductivity, Electrical, an Apparatus for Testing Water by Measurement of its, 538
Congress of Applied Chemistry, Eighth International, 228
Conifers, Oxalic Acid in the Leaves of, Estimation of, 204
Constant Flow Aspirator, 481
————-Pressure Hydrogen Generator, 331
————-Temperature Heating Apparatus for Explosives and Experiments on the Decomposition of Nitrocelluloses, 222
Continuous Fractional Distillation of Water, 474
Copaiba Oil, Examination of, 347
Copper Analysis, the Iodide Method of, the Electrolytic Standardisation of the Conditions of, and Sources of Error, 277

Copper and Salicylic Acid, Colorimetric Estimation of, 104
———, Antimony, Bismuth, and Tin, Electro-Analysis of, with Acidified Chloride Electrolytes, 584
——— -Arsenic Group, Precipitation of, and Separation of its Divisions, 376
———, Determination of: A Modification of the Iodine Method, 66
——— Fungicides, 262
——— in Pyrites, Electro-Analytical Estimation of, 468
———, Iodimetric Estimation of, 375
———, Oxygen and Occluded Gases in, Estimation of, 375
———, Quantitative Separation and Estimation of, by Means of Hydroxylamine Hydrochloride, 583
———, Quantitative Separation of, from Arsenic, Aluminium, Zinc, Tungsten, and Tin, by Means of Hydrogen Peroxide in Presence of Sodium Hydroxide and Sugar, 210
——— -Reducing Power of Normal Urine, 264
——— Reduction Methods, Sugar Analysis by, a Critical Study of, 413
———, Small Quantities of, in Plants, Detection and Estimation of, 313
Cotton Mills, Commercial Starches for Use in, Method for Determining the Value of, 368
——— Wax, Characteristics of, 202
Cow's and Goat's Milk, the Serum of, Refractometer Value of, 317
Cream and Milk Regulations (Draft), Government Report, 109
———, Milk, etc., Certain Proprietary Substances Sold as Preservatives for, Analyses and Methods of Detection of, Report to the Local Government Board on, 155
———, ———, ———, New Preservative for, Note on, 178
Creosote Oil and Other Oils Used for Preserving Timber, Antiseptic Effect of, 56
———, the Sulphonation Test for, Modification of, 269
Cresol, the Volumetric Estimation of, Bromine and Iodine Methods for, 408
p-Cresol and Phenol in the Presence of Each Other, Estimation of, 62
Crucible, Shimer, the Newest form of Determination of Carbon in Steel by Direct Combustion in, with the Aid of a Perforated Clay Disc, 521
Crucibles, Porcelain, etc., Marking of, 289
Crude Fibre, Estimation of, 447
———, the Sweeney Method for, Modification of, 447
Crystallo-Chemical Analysis: A New Method of Chemical Analysis, 526
Cyanides, Detection and Estimation of Halides in the Presence of, by Means of Formaldehyde, 461
Cyanogen and Hydrogen Cyanide, Detection and Determination of, 511
Cypress, the Southern, Oil of, 364

Dairy Federation, the International, Study Committee of, 532
——— Materials and Water, Sterilisation of, by Ultra-Violet Rays, 196
Density of Minerals, Determination of, by Means of Rohrbach Solution of Standard Refractive Index, 148
Densivolumeter, New, for Determination of Density of Solids, 328
Device, Adiabatic, for Bomb Calorimeter, 326
——— for the Combustion of Volatile Substances, Estimation of Halogens in Organic Compounds, Including, 461
Dextrose, Detection of Lævulose in the Presence of, 507
——— in the Presence of Other Optically Active Substances, Estimation of, by Bertrand's Method, 512
——— in Urine and Blood, Estimation of, 140
Diabetic Urine, Glycuronic Acid in, Detection of, 507

Diastatic Power of Malt, 58
Digestive Enzymes, the Assay of, Observations on, 57
——— Value of Papain, Determination of, 405
Digitalis, Chemical Valuation of, 189
Dimethyl-Glyoxime, Action of, on Platinum Salts, 524
Dimethylglyoxime as a Reagent for Ferrous Salts, 102
———, Separation of Nickel and Palladium by Means of, 379
Diphenylamine Test for Gun-Cotton, 512
Direct Determination of Rubber, 574
Dirt in Milk, Estimation of, 450
Dish, the Petri, for Cultivating Fungi and for Biological Analysis, a Substitute for, 561
Distillation Arrangement, Modified Soxhlet Extraction Apparatus with, 592
———, Continuous, Fractional, of Water, 474
——— or Evaporation of Liquids, Apparatus for Controlling, and for Cutting off the Supply of Gas to a Burner after any Desired Length of Time, 221
———, Rapid, of Mercury in Vacuo, Apparatus for, 528
Distilled Vinegar when Stored in Wooden Casks, the Acid-Content of, Alteration of, 23
Drugs, Beverages, and Foods, Saccharin and Dulcin in, Separation and Identification of, 23
Dry Matter in Potatoes, Estimation of, and the Applicability of the Ordinary Tables, 259
Drying Oven, 385
Dulcin and Saccharin in Beverages, Foods, and Drugs, Separation and Identification of, 23
Dyes, Aniline, and Turmeric, in Mustard, Detection of, 557
———, Certain, the Detection and Separation of, on the Application of Adsorption to, 339
Dyestuffs, Azo, Titration of, by Means of Hydrosulphite, 568
———, Coal-Tar, the Seven, Allowed (U.S.A.) to be Employed for Colouring Foodstuffs, Mixtures of, Method for Separating, 19
Dynamite, Exudation of Nitroglycerine from, 572
——— Glycerol, Valuation of, 103

Earth Reactions, Rare, in Non-Aqueous Solvents, 524
Earths, Certain Rare, Fusion of, with Alkali Carbonates. Separation of Tungsten from Iron, Beryllium, and Aluminium, 591
———, the Rare, Separation of, 380
Eastern France, Wines from, Analyses of, 352
Ebullioscopic Determinations at Low Temperatures (−36° to −83° C.), 287
Edible Beans from Hong-Kong, 494
Effluents, Phenols in, Colorimetric Estimation of, 30
———, Sewage, Nitrates in, Estimation of, 149
Eggs, Hens', Composition of, 25
Ehrlich's Reaction for Aldehydes, and Urobilinogen of Urine, 28
Electric Current, Separation of Rennin and Pepsin by Means of the, 141
——— Vacuum Furnace, Determination of Oxygen in Iron and Steel by Reduction in an, 586
Electrical Conductivity, an Apparatus for Testing Water by Measurement of its, 538
——— Furnace for Studying the Course of Reactions under Great Pressure, 288
——— Method, Analysis of Organic Substances by, 331
Electrically-Heated Microscope Slide, 592

Electro-Analysis of Copper, Antimony, Bismuth, and Tin, with Acidified Chloride Constituents, 584
———-Analytical Estimation of Copper in Pyrites, 468
——————— Separation of Metals, Use of the Mercury Cathode, Particularly in, 279
Electrochemical Analysis, Perchloric Acid in, 279
Electrode, the Hydrogen, Rapid Determination of Magnesia in Limestone by Means of, 585
Electrolytes, Acidified Chloride, Electro-Analysis of Copper, Antimony, Bismuth, and Tin with, 584
Electrolytic Apparatus for Use in Invert Sugar Determinations, 576
————— Estimation of Manganese and its Separation from Iron, 419
————— ————— of Molybdenum. Part I., 420
Electrometric Determination of the Reaction of Liquids Containing Carbon Dioxide, 416
Elementary Analysis, 508
Elements, Structural, of Cellular Membranes, 503
————, the Common, System of Qualitative Analysis for. Part V.: Acidic Constituents, 322
Elm-Seed Fat, 201
Embryo and Aleurone Layer of Hordeum, Amyloclastic Secretory Capacities of the, 97
Emulsions, Lecithin, Method of Preparing, and the Subsequent Estimation of their Strength, 307
—————, Oil, Saponin and Saccharin in, Estimation of, 22
Engler's, Redwood's, and Saybolt's Viscosimeters, Comparative Examination of, 223
Enumeration, Direct, of Bacteria in Water by Means of the Ultra-Microscope, Value of, 317
Enzyme Action, Studies on. II. Hydrolytic Action of Some Amino-Acids and Polypeptides on Certain Esters, 353
————, Proteolytic, of Malt, 504
Enzymes, Digestive, the Assay of, Observations on, 57
———— of Honey, Inversion of Sucrose by the, 194
Ergot of Rye, a New Glucoside in: Clavicepsin, 19
Error, Source of, in the Kjeldahl-Gunning Method, 203
———, Sources of, and the Electrolytic Standardisation of the Conditions of the Iodide Method of Copper Analysis, 277
———————, in the Estimation of the Polenske Value of Fats, 256
Essential Oil in Mustard, Estimation of, 309
————— in Preparations of Mustard, Estimation of, 451
Essential Oils, in Spices, etc., Small Quantities of, the Estimation of. Part II., 88
—————, New Philippine, 61
Ester Method for the Detection of Mono-Animo-Acids together with Polypeptides, 272
Esterification of Monamino Acids by Means of Ethyl Iodide. Separation of Pyrrolidonecarboxylic Acid from Glutamic Acid, 360
Esters, Certain, Hydrolytic Action of Some Amino-Acids and Polypeptides on. Studies on Enzyme Action, 353
Ether and Chloroform Vapours in Air, Gasometric Estimation of, 513
———— Petroleum Ether, the Higher Fatty Acids in, Solubilities of the Lead Salts of, 399
——— in Normal Salt Solution, Solubility of, 448
Ethyl Alcohol and Spirits, Methyl Alcohol in, Detection of, 567
Eudiometer, Explosion, Modified, 38
Evacuated Vessel, the Flow of a Gas into, a Convenient Apparatus for Regulating, and for Obtaining an Average Sample of Gas, 250

Evaporation or Distillation of Liquids, Apparatus for Controlling, and for Cutting off the Supply of Gas to a Burner after any Desired Length of Time, 221
Explosion Eudiometer, Modified, 38
Explosions and Fires, Mine, Use of Mice and Birds for Detecting Carbon Monoxide after, 315
Explosives, Constant-Temperature Heating Apparatus for, and Experiments on the Decomposition of Nitrocelluloses, 222
——————, Used in Coal-Mines, Investigations of, 290
Extract in Wort and Beer, Estimation of, 502
Extraction Apparatus, 427
——————-, New, 39
——————-, Soxhlet, Modified, with Distillation Arrangement, 596
—— Thimble, New Form of, 288
—— with Cold Solvents, Estimation of Fat by, 102
Extracts, Flavouring, the Determination of Vanillin in, New Colorimetric Method for, 501
————, Meat, Some New Zealand, Chemical Composition of, 193
——————, the Nitrogenous Constituents of, Changes in the Composition of, 21
————, Tobacco, Nicotine in, Methods of Estimating, 452

Fabrics, Rubberised, Analysis and Testing of, 575
Fat and Lipoids in Blood, Estimation of, and Notes on the So-Called Lipolysis, 561
——, Butter, and Cocoanut Oil in Margarine, the Determination of, Notes on, 122
——————, the Crude Cholesterol of, and Crude Phytosterol of Cocoanut Oil, and the Detection of Cocoanut Oil in Butter Fat, 497
——, Elm-Seed, 201
——, Estimation of, by Extraction with Cold Solvents, 102
——, Hare, Composition of, 255
——, Hydnocarpus, 20
—— in Animal Substances, Kumagawa-Suto Method of Estimating, 354
—— in Cheese, the Estimation of, Modified Butyrometer Tube for, 221
—— in Cocoa, Estimation of, by Means of Zeiss' Refractometer, 495
——, Loss of, during the Drying of Animal Tissues, 355
Fats and Bassia Kernels, 54
—— and Oils, Certain Vegetable, Bromine Adsorption of, 403
——————, Glycerides Occurring in: Heptadecoic Acid and its Glyceride, 411
——, Fractional Saponification of, 558
——, Glycerol in, Estimation of, 571
——, Other, Margarine, and Butter, Benzoic Acid in, Estimation of, 92
——, Saponification of, by Sulphuric Acid, 256
the Polenske Value of, the Estimation of, Sources of Error in, 256
-, Vegetable, Some, Analytical Constants of, 349
Fatty Acid, Free, Effect of, on Specific Gravity of Some Fatty Oils, 410
—— Acids, Small Quantities of, Estimation of the Molecular Weight of, 203
——————, the Higher, in Ether and in Petroleum Ether, Solubilities of the Lead Salts of, 399
—— Oils Obtained from the Seeds of *Illicium Verum* Hook and *Illicium Religiosum* Sieb, Composition of, 495
-, Some, Specific Gravity of, Effect of Free Fatty Acid on, 410
Feeding Materials, Some, Acid-Soluble Phosphorus Compounds of, 193
Fehling's Solution, the Estimation of Furfural by Means of, 41

Fehling's Solution, the Estimation of Reducing Sugars by, Influence of Peptones on, 367
Fermented Beverages, Caramel in, Detection of, 18
Ferric Iron, Presence of Certain Organic Substances, the Estimation of, 179
——- Salts, Compounds of, with Albumoses, 408
Ferricyanides, Reaction of Hydrazine and Hydroxylamine with, and New Methods for Estimating Hydrazine and Ferricyanides, 417
Ferro-Alloys, Carbon in, Use of Oxygen under Pressure for Estimating, 379
Ferrocyanide Titration of Zinc, Influence of Lead on, 585
Ferrocyanides, Estimation of, 362
Ferro-Tungsten, Manganese in, Rapid Estimation of, 107
Ferrotungsten, Tungsten in, Rapid and Exact Estimation of, 220
Ferro-Zircon, Analysis of, 220
————, The Analysis of, a New Method for, and Separation of Zirconium from Iron and Aluminium, 36
Ferrous Ion, Estimation of the, by Means of Iodine, 32
——- Iron in Silicate Rocks, Estimation of, 146
——- Salts, Dimethylglyoxime as a Reagent for, 102
Ferruginous Minerals, Zinc in, Estimation of, 425
Fibre, Crude, Estimation of, 447
——, ——, the Sweeney Method for, Modification of, 447
Fibres, Hemp and Flax, Microscopic Differentiation of, 195
Fibres, Textile, and Woods, Cellulose in, Estimation of, 268, 510
Filter, Inorganic, for Laboratory Purposes, New Type of, 526
—— Pumps, Valve for, 480
Filtration with Alundum Plates, 109
Finishes, Textile, Analysis of, 200
Fires and Explosions, Mine, Use of Mice and Birds for Detecting Carbon Monoxide after, 315
Fish Oils, Viscosity of, 143
Flavouring Extracts, the Determination of Vanillin in, New Colorimetric Method for, 501
Flax and Hemp Fibres, Microscopic Differentiation of, 195
Flour, Baking Qualities of, 190
——, " Gasolene Colour Value " of, the Determination of, Method for, 255
——, Improver, 90
——, Mustard, Analysis of, 55
——, Studies on, 495
——, the Baking Qualities of, Influence of the Concentration of Hydrogen Ions on, 257
——, the Colouring Matter of, the Nature of, and its Relation to Processes of Natural and Artificial Bleaching, Report to the Local Government Board on, 596
Flours, Mustard, and Oil-Cakes, Mustard Oils in, Estimation of, 95
Flue Gases, the Technical Analysis of, Use of the Zeiss Interferometer in, 427
Fluid, Spermatic, Microchemical Reaction of, 358
Fluids, Other Physiological, and Urine, Blood in, Detection of, 506
Fluorescein, Detection of Bromine by, 145
Fluorescence Test for Mineral and Rosin Oils, 30
Fluorides, Detection of, 32
Fluorine in Foods, etc., Detection of, 191
———, Minute Quantities of, in Waters, Minerals, and Living Tissues, Detection and Estimation of, 517
Fluorspar, Valuation of, 210
Food Analysis, New Sublimation Apparatus, and its Application in, 428

Foods and Beverages, Saponin in, Detection of, by Hæmolysis, 350
—— and Drugs Acts, the Sale of, 1875 to 1907, Circular to Local Authorities for the Purposes of, 434
—— AND DRUGS ANALYSIS, ABSTACTS, 18, 51, 92, 137, 189, 254, 306, 347, 400, 446, 492, 557
——, Benzoic Acid in, Detection of, 349
——, Beverages, and Drugs, Saccharin and Dulcin in, Separation and Identification of, 23
——, etc., Cane-Sugar in, Estimation of, 493
——, ——, Fluorine in, Detection of, 191
——, Inspectors of, on the Work of, 71
——, Saccharin in, Estimation of, 194
——, Vegetable, Amounts of Arsenic in, 254
Food-Stuffs and Other Organic Substances, Incineration of, Prior to the Determination of their Phosphorus-Content, 257
————, Phosphorus in, Determination of, by Titration with Uranyl Solutions, 258
Foodstuffs, the Seven Coal-Tar Dyestuffs Allowed (U.S.A.) to be Employed for Colouring, Method for Separating Mixtures of, 19
Formaldehyde, Detection and Estimation of Halides in the Presence of Cyanides by Means of, 461
——, Estimation of, 269
——, Method, Estimation of the Amino-Acids in Plants by Means of the, 312
——, Solutions, the Strength of, Refractometric Estimation of, 202
——, Tetraformal-trisazine from, and Hydrazine Hydrate, a New Reducing Agent for Analytical Use, 369
Formic Acid in Fruit Products, Detection of, 403
—— in Honey, 258
—— in Syrups, Detection and Estimation of, 558
—— in the Presence of Other Acids, Estimation of, by Means of Potassium Permanganate, 270
Formolite Reaction, Application of, to the Analysis of Paraffin Wax, 274
Fractional Distillation of Water, Continuous, 474
—— Saponification of Fats, 558
France, Eastern, Wines from, Analyses of, 352
Free Bromine, Sensitive Reaction of, 581
—— Carbon Dioxide in Water, Estimation of, by Titration with Alkalis in the Presence of Phenolphthaleïn, 582
—— Fatty Acid, Effect of, on Specific Gravity of Some Fatty Oils, 410
—— Lime in Portland Cement, Use of Higher Phenols in Testing for, 582
—— Sulphur, Estimation of, Rapid Volumetric Method for, 151
Freezing-Point of Milk, 307
Frehse's and Vamvaka's Tests, Detection of Saponin and Glycyrrhizin in Lemonades by Means of, 22
Freshness of Milk, Tests for Ascertaining the, 25
Fruit Juice Statistics for the Year 1911, 94
—— Juices, Watered, Nitric Acid in, Detection of, 21
—— Products, Formic Acid in, Detection of, 403
Fulminate, Mercury, Investigations on, 318
Fungi, Cultivating, the Petri Dish for, and for Biological Analysis, a Substitute for, 561
Fungicides, Copper, 262
Furfural, the Estimation of, by Means of Fehling's Solution, 41
Furnace, Electric Vacuum, Determination of Oxygen in Iron and Steel by Reduction in, 586

Furnace, Electrical, for Studying the Course of Reactions under Great Pressure, 288
Fuse and Miners' Squibs, Investigations of, 291

Gas, an Average Sample of, a Convenient Apparatus for Obtaining, and for Regulating the Flow of a Gas into an Evacuated Vessel, 250
——, Analysis Apparatus, New Forms of, 288
——, Apparatus for Cutting off the Supply of, to a Burner after any Desired Length of Time, and for Controlling the Evaporation or Distillation of Liquids, 221
——, Flow of, Combined Governor and Gauge for Regulating, 330
——, Illuminating, Sulphur in, Estimation of, 152
—— Mixtures, Hydrogen and Methane in, Estimation of, 518
————, Rapid Removal of Nitrogen from, by Sparking, Apparatus for, 224
—— Producer, Tar in, Estimation of, 275
—— -Water, Pyridine in the Presence of Ammonia in, Rapid Estimation of, 574
Gases, Evolved, Measurement of, Apparatus for Extracting Liquids with Immiscible Solvents, and for, 69
———, Flue, the Technical Analysis of, Use of the Zeiss Interferometer in, 427
———, Occluded, and Oxygen in Copper, Estimation of, 375
———, Poisonous, from Oilfields, 486
"Gasolene Colour Value" of Flour, the Determination of, Method for, 255
Gasometric Estimation of Ether and Chloroform Vapours in Air, 513
Gauge and Governor, Combined, for Regulating Flow of Gas, 330
Gelatin, Arsenic in, Occurrence of, 94
Gels, Silicic Acid, Vapour Pressure Isothermals of, Apparatus for Determination of, 328
Generator, Hydrogen, Constant-Pressure, 331
Geochemical Interpretation of Water Analysis, 107
Geraniol in Citronella Oil, Direct Estimation of, 363
Gironde, the Department of, Analyses of Wines from, 352
Glands, Thyroid, Iodine Content of, 455
Glasses, Soda Lime, the Refractive Index of, Relation of, to their Chemical Composition, 278
Gliadin or Alcohol-Soluble Protein in Wheat-Flour, Determination of, 19
Glucosamine in Ovomucoid and Pseudomucin, Polarimetric Estimation of, 561
Glucose in Leather, Estimation of, 270
Glucoside, New, in Ergot of Rye: Clavicepsin, 19
Glutamic Acid, Separation of Pyrrolidonecarboxylic Acid from. Esterification of Monamino Acids by Means of Ethyl Iodide, 360
Glyceride, Heptadecoic Acid and its: Glycerides Occurring in Fats and Oils, 411
Glycerol, Dynamite, Valuation of, 103
——— in Fats, Estimation of, 571
——— in Wine, Estimation of, 260
——— in Wines, the Estimation of, New Method for, 351
Glycyrrhizic Acid in Liquorice Juice, Estimation of, 138
Glycyrrhizin and Saponin in Lemonades, Detection of, by Means of Vamvakas' and Frehse's Tests, 22
Glycin Picrate, Composition and Properties of, and Separation of Glycin from Alanin, 570
Glycuronic Acid in Diabetic Urine, Detection of, 507
Goat's and Cow's Milk, the Serum of, Refractometer Value of, 317
Gold Bullion, Sampling, 517
—— Coin, a Counterfeit, Note on, 7
—— Salts, Reaction of, with *m*-Phenylenediamine, 469
——, Silver, and Platinum, Estimation of, 281

Government Reports, 109, 155, 432
Governor and Gauge, Combined, for Regulating Flow of Gas, 330
Granules, Starch, Notes on, and Apparatus for the Estimation of Adulterants in Vegetable Powders, 529
Grass Oils, Aromatic, Part I., 61
———, ———, Part II., 197
———, ———, Part III., 265
Gravimetric Analysis, Simplification of, 31
——— and Volumetric Estimations. The Coagulation of Albumin by Heat and its Precipitation by Mercuric-Potassium Iodide, 458
——————— Methods for the Analysis of Mercury-Ammonium Chloride, 33
Grignard Reagent, Quantitative Estimation of Hydroxy-, Amino-, and Imino-Derivatives of Organic Compounds by Means of, and the Nature of the Changes Taking Place in Solution, 201
Group, the Copper-Arsenic, Precipitation of, and Separation of its Divisions, 376
Guadaloupe Rum, Composition of, 97
Gum in Syrups, Estimation of, 94
—— Resins, Notes on the, 270
—— Tragacanth, Volatile Acidity of, Compared with that of Indian Gum, 318
Gun-Cotton, Diphenylamine Test for, 512
Gutzeit Test, Determination of Small Quantities of Arsenic by the, 579

Hæmolysis, Detection of Saponin in Foods and Beverages by, 350
Hæmolytic Action, their: Toxicity of Mushrooms, 563
Hair of Persons who have Received Subcutaneous Doses of Mercury Compounds, Occurrence of Mercury in. Detection of Minute Quantities of Mercury, 562
Halides in the Presence of Cyanides, Detection and Estimation of, by Means of Formaldehyde, 461
Halogens and Sulphur in Small Quantities of Organic Substances, Estimation of, 216
——— in Organic Compounds, Estimation of, Including a Device for the Combustion of Volatile Substances, 461
———, Other, and Organic Matter, Iodine in the Presence of, Determination of, 418
Hand Photometer, New, 429
Hardy and Arnold, the Method of, for the Rapid Estimation of Sulphur in Siderurgical Products, Modification of, 283
Hare Fat, Composition of, 255
Hawaiian Soil, Humus in, Determination of, 513
Health, Public Act (Regulations as to Food), 1907, Regulations under the, 432
Heat, Alteration of Nitrocellulose and Nitrocellulose Powders by, 104
—— Test for Chinese Wood Oil, 410
Heating Apparatus, Constant-Temperature, for Explosives and Experiments on the Decomposition of Nitrocelluloses, 222
Heats, Latent, of Vaporisation of Mixed Liquids. Part III.: Mixtures of Associated with Non-Associated Liquids, and the Detection of Solvates in Mixtures of Liquids, 362
Heavy Petroleum in Paints and Vegetable Oils, the Detection of, 247
Hemp and Flax Fibres, Microscopic Differentiation of, 195
Hens' Eggs, Composition of, 25
Heptadecoic Acid and its Glyceride: Glycerides Occurring in Fats and Oils, 411
High-Grade Tungsten Alloys, Analysis of, 424
Higher Fatty Acids in Ether and in Petroleum Ether, Solubilities of the Lead Salts of, 399

Higher Oxides of Lead, Analysis of, 148
——— Phenols, Use of, in Testing for Free Lime in Portland Cement, 582
Hippuric Acid, Benzoic Acid, and Phenaceturic Acid in Urine, Estimation of, 356
————— in Urine, the Estimation of, Method for, 507
—————————, ——————, New Method for, 356
Honey Analysis, Application of the Quantitative Precipitin Reaction in, II., 404
———, Formic Acid in, 258
——— from Various Countries, Composition of, 53
———, the Analysis of, Examination of Methods Used in, 449
———, the Enzymes of, Inversion of Sucrose by, 194
———, the Mutarotation of, 95
Hong-Kong, Edible Beans from, 494
Hop, Determination of Hard and Total Soft Resins in the, Comparative study of the Methods for, 191
Hops in Worts and Beers, Bitter Resins of, and their Volumetric Estimation, 139
Hordeum, the Embryo and Aleurone Layer of, Amyloclastic Secretory Capacities of, 97
Horny Tissue and Other Proteins, the Quantity of Tryptophane in, and Colorimetric Method for the Estimation of Tryptophanes, 565
Hübener Bromide Method for the Estimation of Caoutchouc, 509
———————————————— Rubber, 465
Hübener's Bromide Method for the Estimation of Caoutchouc, 202
Humus in Hawaiian Soil, Determination of, 513
———, Soil, Studies on, 466
Husk in Cocoa Powders, Estimation of, 402
Hydnocarpus Fat, 20
Hydrazine and Hydroxylamine, Reaction of, with Ferricyanides, and New Methods for Estimating Hydrazine and Ferricyanides, 417
————, Estimation of, 364
———— Hydrate and Formaldehyde, a New Reducing Agent for Analytical Use, from Tetraformal-trisazine, 369
Hydrocarbon Mixtures, Analysis of, 462
Hydrocarbons, Benzene, and Petroleum Spirit in Turpentine Oil, Estimation of, 274
————— in Beeswax and Carnauba Wax, Estimation of, 348
—————, Other, in Turpentine, Detection of, 311
Hydrochloric Acid, Estimation of Arsenious Acid with Permanganate in Presence of, 417
Hydrochloride, Hydroxylamine, Quantitative Separation and Estimation of Copper by Means of, 583
Hydrocyanic Acid, Rate of Evolution of, from Linseed, under Digestive Conditions, 313
Hydrogen, Active, in Organic Compounds, Quantitative Estimation of, by Magnesium-Methyl Iodide, 571
———— and Methane in Gas Mixtures, Estimation of, 517
———— Cyanide and Cyanogen, Detection and Determination of, 511
———— Electrode, Rapid Determination of Magnesia in Limestone by Means of the, 585
———— Generator, Constant-Pressure, 331
———— Ion Concentrations, the Colorimetric Estimation of, Application of Methyl-Red to, 142
———— Ions in Sea-Water, Concentration of, Measurement of, 425
—————, Influence of the Concentration of, on the Baking Qualities of Flour, 257
———— Peroxide, and Alkali, Behaviour of Invert Sugar in the Presence of, 28

Hydrogen Peroxide and Nitric Acid, Use of a Mixture of, in Analytical Work, 212
—————— in Presence of Sodium Hydroxide and Sugar, Quantitative Separation of Copper from Arsenic, Aluminium, Zinc, Tungsten, and Tin by Means of, 210
———— Sulphide, Qualitative Analysis without, 377
(Hydrogenised) Solidified Oils, 452
Hydrolysis, Complete, of Proteins, Conditions for, 573
Hydrolytic Action of Some Amino-Acids and Polypeptides on Certain Esters. II.: Studies on Enzyme Action, 353
Hydrosulphite, Titration of Azo Dyestuffs by Means of, 568
Hydroxy-, Amino-, and Imino-Derivatives of Organic Compounds, Quantitative Estimation of, by Means of the Grignard Reagent, and the Nature of the Changes Taking Place in Solution, 201
p-Hydroxybenzoic Acid, the Estimation of Small Quantities of Salicylic Acid in Presence of Excess of, Biochemical Method for, 316
β-Hydroxybutyric Acid and Lactic Acid, Estimation of, in Mixtures of the Same, 409
l-*β*-Hydroxybutyric Acid in Urine and Blood, Quantitative Estimation of, 314
Hydroxyl Groups, Alcoholic, and Alcohols, Colour Reaction of, 407
Hydroxylamine and Hydrazine, Reaction of, with Ferricyanides, and New Methods for Estimating Hydrazine and Ferricyanides, 417
—————— Hydrochloride, Quantitative Separation and Estimation of Copper by Means of, 583

Ignition, Effect of, on the Solubility of Soil Phosphates, 523
Illicium Verum Hook and *Illicium Religiosum* Sieb, the Fatty Oils Obtained from the Seeds of, Composition of, 495
Illuminating Gas, Sulphur in, Estimation of, 152
Imino-, Hydroxy-, and Amino-Derivatives of Organic Compounds, Quantitative Estimation of, by Means of the Grignard Reagent, and the Nature of the Changes Taking Place in Solution, 201
Imitation Parchment Papers and "Pergamyn," 144
Immiscible Solvents, Extracting Liquids with, Apparatus for, and for Measurement of Evolved Gases, 69
Improvements in the Ludwig-Sipöcz Method for the Estimation of Water in Silicates, 380
Improver, a Flour, 90
Incineration of Food-Stuffs and Other Organic Substances Prior to the Determination of their Phosphorus-Content, 257
Index, Refractive, of Beeswax, 348
——————, of Chinese Wood Oil, 410
——————, of Soda Lime Glasses, Relation of, to their Chemical Composition, 278
Indian Gum, Volatile Acidity of Gum Tragacanth Compared with that of, 318
Indicator in Iodimetric Titrations, the Use of Methylene Blue as, 252
————, Use of Phenolphthaleïn as an, 320
Indigos Containing Starch, Analysis of, 319
Indole Reaction, the, 562
INORGANIC ANALYSIS, ABSTRACTS, 31, 64, 106, 145, 206, 276, 321, 372, 414, 468, 516, 579
———— Filter for Laboratory Purposes, New Type of, 526
———— Phosphorus in Plant Substances, Estimation of, 472
INSTITUTE OF CHEMISTRY, 76, 119, 162, 294, 438
Intensity Distribution in Blood Spectra, Photographic Estimation of, 313

Interferometer, New, for Liquids, Estimation of the Concentration of Colloidal Solutions with, 382
——————, the Zeiss, Use of, in the Technical Analysis of Flue Gases, 427
Invert Sugar Determinations, Electrolytic Apparatus for Use in, 576
——————, Expanded Meissl-Hiller Table for, 575
—————— in the Presence of Alkali and Hydrogen Peroxide, Behaviour of, 28
Iodide, Cadmium Potassium, Determination of Ozone by Means of, 587
———, Magnesium Methyl, Quantitative Estimation of Active Hydrogen in Organic Compounds by, 571
———, Method of Copper Analysis, the Electrolytic Standardisation of the Conditions of, and Sources of Error, 277
Iodides, Determination of, by Direct Titration, 378
———, etc., the Method of Precipitating Metals as, from Sulphuric Acid Solution, Applications of, 519
———, the Estimation of, Methods for, 519
Iodimetric Estimation of Copper, 375
————— Titrations, Methylene Blue as an Indicator in, the Use of, 252
Iodine and Bromine Methods for the Volumetric Estimation of Cresol, 408
——— Content of Thyroid Glands, 455
———, Estimation of the Ferrous Ion by Means of, 32
——— in the Presence of Other Halogens and Organic Matter, Determination of, 418
——— Method, a Modification of the: Determination of Copper, 66
——— Value and the Structure of Acids of the Oleic Series, Relationship between, 463
Ion, the Ferrous, Estimation of, by Means of Iodine, 32
Ions, Hydrogen, in Sea-Water, Concentration of, Measurement of, 425
——————, Influence of the Concentration of, on the Baking Qualities of Flour, 257
Iron and Aluminium, Separation of Zirconium from, and a New Method for the Analysis of Ferro-Zircon, 36
—— and Steel, Oxygen in, Determination of, by Reduction in an Electric Vacuum Furnace, 586
——————, Paint Films and Preservative Coatings for, New Method for Testing, 213
——————, Sulphur in Volumetric Estimation of, 67
——, Beryllium, and Aluminium, Separation of Tungsten from. Fusion of Certain Rare Earths with Alkali Carbonates, 591
——, Electrolytic Estimation of Manganese, and its Separation from, 419
——, Ferric, in Presence of Certain Organic Substances, the Estimation of, 179
——, Ferrous, in Silicate Rocks, Estimation of, 146
——, in Lead and its Oxides, Colorimetric Determination of, 520
——, in Milk, Estimation of, 308
—— in (Pharmaceutical) Scale Preparations, Determination of, 306
——, Separation of, from Titanium, 323, 469
——, Small Quantities of, in Organic Substances, Estimation of, 32
Isothermals, Vapour Pressure, of Silicic Acid Gels, Apparatus for Determination of, 328

Japanese and Chinese Wood Oil, the Examination of, 543
Juice, Liquorice, Glycyrrhizic Acid in, Estimation of, 138
Juices, Fruit, Watered, Detection of Nitric Acid in, 21
———, Plant, the Oxidase Content of, Measurement of, 261

Ketoses and Aldoses, Method of Distinguishing between, 266

Kirschner and Polenske Values for Margarines Containing Cocoanut or Palm Kernel Oils, on the Relation between, 183
Kjeldahl-Gunning Method, Source of Error in the, 203
Knapp and Shrewsbury's Process for Estimating Cocoanut Oil, Notes on, 3
Kola Nuts from British West Africa, 258
Kumagawa-Suto Method of Estimating Fat in Animal Substances, 354

Laboratory Apparatus, Two New Pieces of, 528
——— Centrifuge for Quantitative Separation and Estimation of Solids and Liquids, 286
———, Government, the Work of, for the Year Ending March 31, 1912, Report of the Government Chemist upon, with Appendices, 530
——— Purposes, Inorganic Filter for, New Type of, 526
Studies of Rennin, 405
Lactic Acid, Analysis of,29
———, and β-Hydroxybutyric Acid, Estimation of, in Mixtures of the Same, 409
——— in the Presence of Proteins, Estimation of, 403
(Lactose, a Correction for), Reducing Sugar Methods, Unification of, 145
———, the Estimation of, a New Method for, and Behaviour of Milk when Treated with Ammonium Sulphate, 350
Lævulose, Detection of, in the Presence of Dextrose, 507
——— Reaction, the Seliwanoff, Physico-Chemical Basis of, 367
Lamps, Sodium Flame, for Polarisation, 594
Landsberger Boiling-Point Apparatus, Modification of, 326
Lanthanum, Quantitative Separation of, from Yttrium, 469
Lard and Butter, Cocoanut Oil in, Detection of, 93
Latent Heats of Vaporisation of Mixed Liquids. Part III.: Mixtures of Associated with Non-Associated Liquids, and the Detection of Solvates in Mixtures of Liquids, 362
Lead and its Oxides, Iron in, Colorimetric Determination of, 520
——— Arsenates, Commercial, Determination of Soluble Arsenic in, Short Method for, 206
——— Assay, the Ammonium Molybdate Method of, on the Effect of Lime on, 242
———, Higher Oxides of, Analysis of, 148
———, Influence of, on the Ferrocyanide Titration of Zinc, 585
——— Salts of the Higher Fatty Acids in Ether and in Petroleum Ether, Solubilities of, 399
Leather, Glucose in, Estimation of, 270
Leaves of Conifers, Oxalic Acid in, Estimation of, 204
———, Uva-Ursi, the Analysis of, Application of the Biochemical Method for, 24
Lecithin Emulsions, Method of Preparing, and the Subsequent Estimation of their Strength, 307
——— in Medicinal Tablets, Estimation of, 449
Lemon Peel, Candied, Analysis of, 20
Lemonades, Saponin and Glycyrrhizin in, Detection of, by Means of Vamvakas' and Frehse's Tests, 22
Leucocyte, Catalase, and Reductase Tests, Comparative Value of, in Detecting Mastitis, 401
Light, Catalytic Action of, on the Oxidation of Phenolphthalin to Phenolphthaleïn, 30
——— Energy, Application of, to the Study of Certain Problems in Analytical Chemistry, 478
Lime, Free, in Portland Cement, Use of Higher Phenols in Testing for, 582
——— Liquors, Sulphides in, Estimation of, 325

Lime, On the Effect of, on the Ammonium Molybdate Method of Lead Assay, 242
Limestone, Rapid Determination of Magnesia in, by Means of the Hydrogen Electrode, 585
Linseed, Evolution of Hydrocyanic Acid from, under Digestive Conditions, Rate of, 313
——— Oil and its Adulterants, Tests for, 192
Lintner's Polarimetric Method for the Determination of Starch, 498
Lipase, Castor-Bean, Experiments with, 316
Lipoids, and Fat in Blood, Estimation of, and Notes on the So-Called Lipolysis, 561
———, Complex, Proximate Analysis of, by Alcoholysis, 463
Lipolysis, the So-Called, Notes on, and Estimation of Fat and Lipoids in Blood, 561
Liqueur, and Cherries, Maraschino, Benzaldehyde in, Estimation of, 446
Liquids and Solids, Quantitative Separation and Estimation of, Laboratory Centrifuge for, 286
———, Apparatus for Controlling the Evaporation or Distillation of, and for Cutting off the Supply of Gas to a Burner after any Desired Length of Time, 221
———, Automatic Analysis of, Apparatus for, 480
———, Boiling, the Bumping of, Device for Preventing, 153
———, Containing Carbon Dioxide, the Reaction of, Electrometric Determination of, 416
———, Extracting with Immiscible Solvents, Apparatus for, and for Measurement of Evolved Gases, 69
———, Mixed, Vaporisation of, Latent Heats of. Part III.: Mixtures of Associated with Non-Associated Liquids, and the Detection of Solvates in Mixtures of Liquids, 362
——— in an Atmosphere Free from Carbon Dioxide, Apparatus for Measuring, 222
———, the Boiling-Points of which Lie Close together, or which Form Mixtures of Minimum Boiling-Point, Separation of, 272
———, the New Interferometer for, Estimation of, the Concentration of Colloidal Solutions with, 382
———, Volatile, Molecular Weights of, Determination of, 595
-- Organic, Combustion of, 361
Liquorice Juice, Glycyrrhizic Acid in, Estimation of, 138
Liquors, Lime, Sulphides in, Estimation of, 325
Lithium, Determination of, 470
Lithopone, the Analysis of, 238
Living Tissues, Waters, and Minerals, Minute Quantities of Fluorine in, Detection and Estimation of, 517
Lucidol (Benzoyl Peroxide), Influence of, on Certain Qualitative Tests for Oils, 273
Ludwig-Sipöcz Method for the Estimation of Water in Silicates, Improvements in, 380
"Luminator" Treatment of Water, 154
Luminescence Analysis, 526

Magnesia, in Admixture with Magnesium Carbonate, Estimation of, 211
——— in Limestone, Rapid Determination of, by Means of the Hydrogen Electrode, 585
——— Rods, Use of, Instead of Platinum Wire, in Analytical Work, 222
Magnesium Carbonate, Magnesia in Admixture with, Estimation of, 211
———, Gravimetric Estimation of, 471
——— Methyl Iodide, Quantitative Estimation of Active Hydrogen, in Organic Compounds by, 571
Malic Acid, Determination of, 29
——— and Succinic Acids in Wine, Estimation of, 453
Malt, Proteolytic Enzyme of, 504

Malt, the Diastatic Power of, 58
—— Vinegar, Standards for, 123
Manganese, Bismuthate Method for, 148
————, Determination of, by the Bismuthate Method, 522
————, Electrolytic Estimation of, and its Separation from Iron, 419
————, in Ferro-Tungsten, Rapid Estimation of, 107
————, the Estimation of, by the Bismuthate Method, 554
————, Total, in Soils, Estimation of, 423
————, Volumetric Estimation of, 471
Maraschino Cherries and Liqueur, Benzaldehyde in, Estimation of, 446
Margarine, Butter, and Other Fats, Benzoic Acid in, Estimation of, 92
————, Butter Fat and Cocoanut Oil in, the Determination of, Notes on, 122
Margarines Containing Cocoanut or Palm Kernel Oils, the Kirschner and Polenske Values for, on the Relation between, 183
Mastitis, Comparative Value of the Catalase, Reductase, and Leucocyte Tests in Detecting, 401
Meal, Examination of, 139
Measurement of Concentration of Hydrogen Ions in Sea-Water, 425
———— of Evolved Gases, Apparatus for, and for Extracting Liquids with Immiscible Solvents, 69
———— of Relative Tryptic Activity, 457
Measuring Absolute Viscosity, Method of, 154
———— Liquids in an Atmosphere Free from Carbon Dioxide, Apparatus for, 222
Meat Extracts, Some New Zealand, Chemical Composition of, 193
————, the Nitrogenous Constituents of, Changes in the Composition of, 21
Meats, Potassium Nitrate in, Estimation of, 140
———, the Changes Taking Place in, Physico-Chemical Method for Determining, 455
Mechanical Analysis of Arid Soils, 588
———— Soil Analysis, Modification of the Method of, 589
Medicinal Tablets, Lecithin in, Estimation of, 449
Meissl-Hiller Table, Expanded, for Invert Sugar, 575
Melting-Point, Determination of, with the Aid of the Microscope, 428
———— of Pitch, New Nethod for Determining, 62
Membranes, Cellular, Structural Elements of, 503
Mercuric-Potassium Iodide, the Coagulation of Albumin by Heat and its Precipitation by, Gravimetric and Volumetric Estimations, 458
Mercurisalicylic Acid, and Oxyphenyldimercuric Acetate, Mercury in, Estimation of, 103
Mercurous Nitrate and Potassium Iodide as Sensitive Reagents for Tungsten and Molybdenum, 378
Mercury-Ammonium Chloride, the Analysis of, Gravimetric and Volumetric Methods for, 33
———— Cathode, Use of, Particularly in the Electro-Analytical Separation of Metals, 279
———— Compounds, Persons who have Received Subcutaneous Doses of, Occurrence of Mercury in the Hair of, Detection of Minute Quantities of Mercury, 562
————, Estimation of, Use of Arsenious Acid in Volumetric Analysis, 321
———— Fulminate, Investigations on, 318
———— in Oxyphenyldimercuric Acetate and Mercurisalicylic Acid, Estimation of, 103
———— in Smokeless Powder, Estimation of, 273
———— in Vacuo, Rapid Distillation of, Apparatus for, 528
———— Thread, Beckmann Thermometer with a Modified Arrangement for Setting the, 429

Metallic Carbonates, Influence of, on the Estimation of the Volatile Substances in Coal, 360
Metals, Other, and Antimony, Separation of Arsenic from, by Means of Methyl Alcohol in a Current of Air, 414
——, ——, ————, the Separation of Arsenic from, with Some Applications to Toxicological Work, 229
——, the Electro-Analytical Separation of, Use of the Mercury Cathode, particularly in, 279
——, the Method of Precipitating, as Iodides, etc., from Sulphuric Acid Solution, Applications of, 519
Methane and Hydrogen in Gas Mixtures, Estimation of, 518
Methyl Alcohol, Detection and Estimation of, 364
———— in a Current of Air, Separation of Arsenic from Antimony and Other Metals by Means of, 414
———— in Admixture with Alcohol, and Especially in Spirits, Estimation of, 514
———— in Ethyl Alcohol and Spirits, Detection of, 567
————, Small Quantities of, Note on the Determination of, 16
—— -Red, Application of, to the Colorimetric Estimation of Hydrogen Ion Concentrations, 142
Methylcyclopentenolon in Pyroligneous Acid, Occurrence of, 319
Methylene Blue as an Indicator in Iodimetric Titrations, The Use of, 252
Metropolitan Water Board, Research Work of, Seventh Report on, 195
Mice and Birds, Use of, for Detecting Carbon Monoxide after Mine Fires and Explosions, 315
Micro-Chemical Detection of Certain Alkaloids, 137
———— Reaction of Spermatic Fluid, 358
———— Tests for the Identification of Some of the Alkaloids, 400
Microscope, Determination of Melting-Point with the Aid of the, 428
——— Slide, Electrically-Heated, 592
Milk and Cream Regulations (Draft), Government Report, 109
——, Australian (Victorian), Composition of, 47
——, Benzoic Acid in, the Detection of, Note on, 346
——, Condensed, Sucrose in, Estimation of, 450
——, Cow's and Goat's, the Serum of, Refractometer Value of, 317
——, Cream, etc., Certain Proprietary Substances Sold as Preservatives for, Analyses and Methods of Detection of, Report to the Local Government Board on, 155
——, ——, ——, New Preservative for, Note on, 178
——, Dirt in, Estimation of, 450
——, Freezing-Point of, 307
——, Iron in, Estimation of, 308
——, Serums, Preparation of, and their Use in the Analysis of Milk, 450
——, Sweetened Condensed, Australian, Composition of, 49
——, the Alkalinity and Peroxydase of, are they Identical? 141
——, the Composition of, 298
——, the Freshness of, Tests for Ascertaining, 25
——, the Reductase Test, Acidity, and Alcohol Test in, Relations between, 26
——, the Souring of, 168
——, when Treated with Ammonium Sulphate, Behaviour of, and a New Method for the Estimation of Lactose, 350
Mills, Cotton, Commercial Starches for Use in, Method for determining the Value of, 368
Mimosa Flowers, Tincture of, Detection of Minute Traces of Boric Acid by Means of, 558

Mine Fires and Explosions, Use of Mice and Birds for Detecting Carbon Monoxide after, 315
Mineral and Rosin Oils, Fluorescence Test for, 30
——— Oils, Pitch, etc., Asphalt in, Estimation of, 58
Minerals and Rocks, Radium in, Estimation of, 323, 421
———, Ferruginous, Zinc in, Estimation of, 425
———, the Density of, Determination of, by Means of Rohrbach Solution of Standard Refractive Index, 148
———, Waters, and Living Tissues, Minute Quantities of Fluorine in, Detection and Estimation of, 517
Miners' Squibs and Fuse, Investigations of, 291
Mkonga Oil, 256
Modified Form of Stability Test, 573
Moist Combustion, Estimation of Carbon by, 459
Moisture, Estimation of, Calcium Carbide Method for, 475
Molasses, Water in, Determination of, 560
Molecular Weight of Small Quantities of Fatty Acids, Estimation of, 203
——— Weights of Volatile Liquids, Determination of, 595
Molybdenum and Tungsten, Sensitive Reagents for, Potassium Iodide and Mercurous Nitrate as, 378
———, Electrolytic Estimation of. Part I., 420
Monamino Acids, Esterification of, by Means of Ethyl Iodide. Separation of Pyrrolidonecarboxylic Acid from Glutamic Acid, 360
Monazite Sand, Thorium, Especially in, Quantitative Estimation of, by Means of Sodium Hypophosphate, 423
Mono-Amino-Acids, together with Polypeptides, the Detection of, Ester Method for, 272
Morphine in Opiates, Estimation of, 308
Mushrooms, Toxicity of: Their Hæmolytic Action, 563
Mustard, Aniline Dyes and Turmeric in, Detection of, 557
———, Essential Oil in, Estimation of, 309
——— Flour, Analysis of, 55
——— Flours and Oil-Cakes, Mustard Oils in, Estimation of, 95
———, Preparations of, Essential Oil in, Estimation of, 451
Mutarotation of Honey, 95

Natural and Petroleum-Residue Asphalts, Differentiation between, 407
Neo- and Allo-Chlorophyll, in the Presence of One Another, Methods for Determining, 460
Neutral Ammonium Citrate Solution, 372
——— Salt, and Alcohol, Simultaneous Effect of, on the Sensitiveness of Phenolphthalein, 321
New Zealand Meat Extracts, Some, Chemical Composition of, 193
Nickel and Palladium, Separation of, by Means of Dimethylglyoxime, 379
——— and Zinc, the Gravimetric Separation of, 443
——— Baths, Boric Acid in, Estimation of, 277
Nicotine in Tobacco, Estimation of, 311
——— Extracts, Methods of Estimating, 452
Nitrate, Mercurous, and Potassium Iodide, as Sensitive Reagents for Tungsten and Molybdenum, 378
——— of Soda, Rational Analysis of, 523
Nitrates and Nitrites in Water, Estimation of, by Schlœsing's Method, 212
——— in Presence of Nitrites, Detection of, 149
——— in Sewage Effluents, Estimation of, 149

Nitrating and Waste Acids, Sulphuric Acid, Nitric Acid, and Nitrous Acid in, Volumetric Estimation of, 381
Nitric Acid and Hydrogen Peroxide, Use of a Mixture of, in Analytical Work, 212
————, the Separation of Nitrous Acid from, and the Estimation of Nitrites, New Volumetric Method for, 586
———— in Acetic Acid Solution, the Stability of. The Determination of Nitric and Nitrous Acids in Acetic Acid Solution, 303
———— in Watered Fruit Juices, Detection of, 21
—— and Nitrous Acids in Acetic Acid Solution, the Determination of. The Stability of Nitric Acid in Acetic Acid Solution, 303
Nitrites and Nitrates in Water, Estimation of, by Schlœsing's Method, 212
———, Nitrates in Presence of, Detection of, 149
———, the Estimation of, and the Separation of Nitrous Acid from Nitric Acid, New Volumetric Method for, 586
Nitrocellulose and Nitrocellulose Powders, Alteration of, by Heat, 104
-, Sulphur in, Estimation of, 152, 273
—— Powders, New Stability Test for, 572
Nitrocelluloses, Experiments on the Decomposition of, and Constant-Temperature Heating Apparatus for Explosives, 222
Nitro-Compounds, Separation of Nitroglycerine from, 572
Nitrogen, Organic, Activity of, as Measured by the Alkaline Permanganate Method, 365
———, Rapid Removal of, from Gas Mixtures by Sparking, Apparatus for, 224
Nitrogenous Constituents of Meat-Extracts, the Composition of Changes in, 21
Nitroglycerine, Exudation of, from Dynamite, 572
—————, Separation of, from Nitro-Compounds, 572
—————, the Determination of, by Means of the Nitrometer, Effect of the Nitrotoluenes on, 572
Nitrous Acid, New Reaction of, 33
—————, Nitric Acid, and Sulphuric Acid, in Nitrating and Waste Acids, Volumetric Estimation of, 381
—————, Separation of, from Nitric Acid, and the Estimation of Nitrites, New Volumetric Method for, 586
—————, Small Quantities of, the Detection and Estimation of, a New Method for, 345
——— and Nitric Acids in Acetic Acid Solution, the Determination of, the Stability of Nitric Acid in Acetic Acid Solution, 303
Nitrotoluenes, Effect of, on the Determination of Nitroglycerine by Means of the Nitrometer, 572
Non-Aqueous Solvents, Rare Earth Reactions in, 524
Normal Carbonate, Much, Detection of Small Quantities of Alkali Bicarbonate in Presence of, 372
——— Salt Solution, Solubility of Ether in, 448
——— Urine, Combined Glycuronic Acid in, Detection of, 562
—————, Copper-Reducing Power of, 264
Nutrition, Animal, the Value of Ordinary and Treated Sawdust for, Experiments on, 559
Nuts, Kola, from British West Africa, 258
——, Shea-, and Shea-Butter, 496

OBITUARY NOTICES:
Divers, Edward, M.D., F.I.C., F.R.S., 167
Ekins, Arthur Edward, J.P., F.I.C., 121, 165
Muter, Dr. John, F.I.C., 41, 77

OBITUARY NOTICES—*continued*
Newlands, Benjamin Edward Reina, 391
Pattinson, John, J.P., F.I.C., 121, 166
Sanford, Percy Gerald, 2
Occluded Gases and Oxygen in Copper, Estimation of, 375
Oil and Cake, Tea-Seed, 500
—, Arachis, the Detection and Estimation of, 487, 537
—-Cakes and Mustard Flours, Mustard Oils in, Estimation of, 95
—, Chinese and Japanese Wood, the Examination of, 543
—, ——— Wood, Heat Test for, 410
—, —————, Method of Examining, 410
—, —————, Refractive Index of, 410
—, Citronella, Geraniol in, Direct Estimation of, 363
—, Cocoanut, and Butter Fat in Margarine, the Determination of, Notes on, 122
—, ————, Crude Phytosterol of, and the Crude Cholesterol of Butter Fat, and the Detection of Cocoanut Oil in Butter Fat, 497
—, ————, in Butter and Lard, Detection of, 93
—, ————, Shrewsbury and Knapp's Process for Estimating, Notes on, 3
—, Copaiba, Examination of, 347
—, Creosote, and Other Oils Used for Preserving Timber, Antiseptic Effect of, 56
—, Emulsions, Saponin and Saccharin in, Estimation of, 22
—, Essential, in Mustard, Estimation of, 309
—, Linseed, and its Adulterants, Tests for, 192
—, Mkonga, 256
— of Citronella, Analysis of, 567
— of Savin, the Constituents of, 295
— of the Southern Cypress, 364
— of Turpentine, the Purity of, Tests for Determining, 371
—, Palm, from Different Varieties of Palm, Characteristics of, 55
—, Pine, in Turpentine Oil, Detection of, 143
—, Savin, Detection of, in Toxicological Cases, 357
—, Sesame, in Other Oils, Reaction for Detecting, 62
—, Turpentine, Detection of Pine Oil in, 143
—, —————, Petroleum Spirit and Benzene Hydrocarbons in, Estimation of, 274
Oilfields, Poisonous Gases from, 486
Oils and Fats, Certain Vegetable, Bromine Absorption of, 403
——————, Glycerides Occurring in: Heptadecoic Acid and its Glyceride, 411
——, Carbon Bisulphide in, Detection of, 101
——, Certain Qualitative Tests for, Influence of Lucidol (Benzoyl Peroxide) on, 273
——, Champaca, 28
——, Cocoanut or Palm Kernel, Margarines Containing, on the Relation between the Kirschner and Polenske Values for, 183
——, Essential, in Preparations of Mustard, Estimation of, 451
——, ————, New Philippine, 61
——, ————, Small Quantities of, in Spices, etc., the Estimation of, Part II., 88
——, Fatty, Obtained from the Seeds of *Illicium Verum* Hook and *Illicium Religiosum* Sieb, Composition of, 495
——, Fatty, Some, Specific Gravity of, Effect of Free Fatty Acid on, 410
——, Fish, Viscosity of, 143
——, Grass, Aromatic, Part I., 61
——, ——, ————, Part II., 197
——, ——, ————, Part III., 265
——, (Hydrogenised), Solidified, 452
——, Mineral and Rosin, Fluorescence Test for, 30

Oils, Mineral, Pitch, etc., Asphalt in, Estimation of, 58
——, Mustard, in Oil-Cakes and Mustard Flours, Estimation of, 95
——, New Oxygen Absorption Method for, 412
——, Other, and Creosote Oil, Used for Preserving Timber, Antiseptic Effect of, 56
——, Other, Sesame Oil in, Reaction for Detecting, 62
——, Palm Kernel or Cocoanut, Margarines Containing, on the Relation between the Kirschner and Polenske Values for, 183
——, Rosin and Mineral, Fluorescence Test for, 30
——, Vegetable, and Paints, Heavy Petroleum in, the Detection of, 247
——, Vegetable, Serger's Reaction for, Notes on, 412
——, Volatile, Cineol in, Estimation of, 461
Oleic Series, Acids of the, Relationship between the Iodine Value and the Structure of, 463
Opiates, Morphine in, Estimation of, 308
Optical Method, New, for the Estimation of the Various Proteins in Ox Serum: The Proteins of Ox Serum. Refractive Indices of Solutions of Certain Proteins, 355
Ores and Bullion, Small Amounts of Platinum in, Direct Determination of, 281
——, Steels, etc., Determination of Vanadium in, Rapid Method for, Based on its Quantitative Inclusion by the Phosphomolybdate Precipitate, 284
ORGANIC ANALYSIS, ABSTRACTS: 28, 58, 100, 142, 197, 265, 317, 359, 407, 458, 508, 565
———— Bases, New Reaction for, 319
———— Compounds, Arsenic in, Detection and Estimation of, 66, 360
———— ————, ————, Estimation of, 579
———— ————, Estimation of Halogens in, Including a Device for the Combustion of Volatile Substances, 461
———— ————, Hydroxy-, Amino-, and Imino- Derivatives of, Quantitative Estimation of, by Means of the Grignard Reagent, and the Nature of the Changes Taking Place in Solution, 201
———— ————, Quantitative Estimation of Active Hydrogen in by Magnesium-Methyl Iodide, 571
———— ————, the Relative Reactivity of, Method for Determining, 204
———— Liquids, Volatile, Combustion of, 361
———— Matter, and Other Halogens, Iodine in the Presence of, Determination of, 418
———— Nitrogen, Activity of, as Measured by the Alkaline Permanganate Method, 365
———— Substances, Action of Permanganate on, 366
———— ————, Analysis of, by an Electrical Method, 331
———— ————, Certain, the Estimation of Ferric Iron in Presence of, 179
———— ————, Other, and Food-Stuffs, Incineration of, Prior to the Determination of their Phosphorus-Content, 257
———— ————, ——, ————, Phosphorus in, Determination of, by Titration with Uranyl Solutions, 258
———— ————, Small Quantities of, Estimation of Sulphur and of Halogens in, 216
———— ————, Small Quantities of Iron in, Estimation of, 32
Oven, Drying, 385
Ovomucoid and Pseudomucin, Glucosamine in, Polarimetric Estimation of, 561
Ox Serum, the Proteins of: A New Optical Method for the Estimation of the Various Proteins in Ox Serum. Refractive Indices of Solutions of Certain Proteins, 355
Oxalic Acid in the Leaves of Conifers, Estimation of, 204
———— Vegetable Substances, Estimation of, 564

Oxidase Content of Plant Juices, Measurement of, 261
Oxidation of Phenolphthalin to Phenolphthaleïn, Catalytic Action of Light on, 30
Oxides, Higher, of Lead, Analysis of, 148
———, Lead and its, Iron in, Colorimetric Determination of, 520
Oxygen Absorption Method, New, for Oils, 412
——— and Occluded Gases in Copper, Estimation of, 375
——— in Iron and Steel, Determination of, by Reduction in an Electric Vacuum Furnace, 586
——— under Pressure, Use of, for Estimating Carbon in Ferro-Alloys, 379
Oxyphenyldimercuric Acetate and Mercurisalicylic Acid, Mercury in, Estimation of, 103
Ozone, Absorption of Ultra-Violet Rays in, and its Application to the Estimation of Ozone in Small Concentrations, 472
———, Determination of, by Means of Cadmium Potassium Iodide, 587

Paint Films and Preservative Coatings for Iron and Steel, New Method for Testing, 213
Paints and Vegetable Oils, Heavy Petroleum in, the Detection of, 247
Palladium and Nickel, Separation of, by Means of Dimethylglyoxime, 379
Palm, Kernel or Cocoanut Oils, Margarines Containing, on the Relation between the Kirschner and Polenske Values for, 183
—— Oil from Different Varieties of Palm, Characteristics of, 55
Pancreas Function, the Quantitative Investigation of, Modification of Wohlgemuth's Method for, 315
Pancreatic Amylase, a Further Investigation of the Properties of Studies of Amylases, 454
Pancreatin, the Tryptic Value of, New Method for Determining, 26
Papain, the Digestive Value of, Determination of, 405
Paraffin Wax and Ceresin, Mixtures of, Analysis of, 59
————, the Analysis of, Application of the Formolite Reaction to, 274
Paraldehyde, Acetaldehyde in, Estimation of, 265
Parchment Paper, 465
———— Papers, Imitation, and "Pergamyn," 144
Peel, Candied Lemon, Analysis of, 20
Pepper, Cayenne, Adulteration of, Detection of, 259
Pepsin and Rennin, Separation of, by Means of the Electric Current, 141
Peptones, Influence of, on the Estimation of Reducing Sugars by Fehling's Solution, 367
Perchlorates, Certain, Perchloric Acid in, Estimation of, 33
————, Quantitative Determination of, 374
Perchloric Acid in Electrochemical Analysis, 279
"Pergamyn" and Imitation Parchment Papers, 144
Permanganate, Action of, on Organic Substances, 366
————, Estimation of Arsenious Acid with, in Presence of Hydrochloric Acid, 417
———— Method, the Alkaline, Activity of Organic Nitrogen as Measured by, 365
————, Potassium, Estimation of Formic Acid in the Presence of Other Acids by Means of, 270
———— Solution, Potassium, Standardisation of, by Sodium Oxalate, 280
Peroxide, Hydrogen, and Alkali, Behaviour of Invert Sugar in the Presence of, 28
Peroxydase and Alkalinity of Milk; Are they Identical? 141
————, Nature of, 504
————, Separation of, from Catalase, 315
Peru Balsam, Cinnamein in, Estimation of, 93

Pétri Dish for Cultivating Fungi and for Biological Analysis, a Substitute for the, 561
Petroleum and its Products, Water in, Determination of, 573
——— Ether and Ether, the Higher Fatty Acids in, Solubilities of the Lead Salts of, 399
———, Heavy, in Paints and Vegetable Oils, the Detection of, 247
——— in Turpentine, Estimation of, 371
———, Mixtures Containing, the Examination of, 8
——— -Residue and Natural Asphalts, Differentiation Between, 407
——— Residues, "Acid Resins" from, 515
——— Spirit and Benzene Hydrocarbons in Turpentine Oil, Estimation of, 274
———, Sulphur in, Estimation of, 217
Petroleums of the San Joaquin Valley of California, Physical and Chemical Properties of, 104
Phenaceturic Acid, Benzoic Acid, and Hippuric Acid in Urine, Estimation of, 356
Phenol and *p*-Cresol in the Presence of Each Other, Estimation of, 62
——— in Urine, Estimation of, 356
——— *p*-Sulphonic Acid, the Determination of, Volumetric Method for, 464
———, Rapid Methods for Determining, 515
Phenolphthaleïn, as an Indicator, Use of, 320
———, Estimation of Free Carbon Dioxide in Water by Titration with Alkalis in the Presence of, 582
———, the Oxidation of Phenolphthalin to, Catalytic Action of Light on, 30
———, the Sensitiveness of, Simultaneous Effect of Alcohol and a Neutral Salt on, 321
Phenols, Higher, Use of, in Testing for Free Lime in Portland Cement, 582
———, in Effluents, Colorimetric Estimation of, 30
m-Phenylenediamine, Reaction of Gold Salts with, 469
Philippine Essential Oils, New, 61
Phosphates, Soil, the Solubility of, Effect of Ignition on, 523
Phosphomolybdate Estimation of Phosphoric Acid in Soils, 130
——— Precipitate, Rapid Method for Determination of Vanadium in Steels, Ores, etc., Based on its Quantitative Inclusion by the, 284
Phosphomolybdates, Separation of, from Silicomolybdates, 107
Phosphoric Acid, Estimation of, by Von Lorenz's Method, 149
———, in Presence of Colloidal Silicic Acid, Estimation of, 214
——— in Soils, the Phosphomolybdate Estimation of, 130
——— in Wines, Estimation of, 453
Phosphorus Compounds, Acid-Soluble, of Some Feeding Materials, 193
——— in Food-Stuffs and Other Organic Substances, Determination of, by Titration, with Uranyl Solutions, 258
——— in Wine, Estimation of, 24
———, Inorganic, in Plant Substances, Estimation of, 472
Phosphotungstic Acid, Blue Colour-Reaction of, with Uric Acid and Other Substances, 355
Photographic Estimation of the Intensity Distribution in Blood Spectra, 313
Photometer, New Hand, 429
Physical Analysis of Soils, New Method for, 36
Physico-Chemical Basis of the Seliwanoff Lævulose Reaction, 367
——— Method for Determining the Changes taking Place in Meats, 455
——— Volumetric Method, Estimation of Sulphates in Solution by, 282
Physiological Fluids, Other, and Urine, Blood in, Detection of, 506
Phytosterol, Crude, of Cocoanut Oil, and the Crude Cholesterol of Butter Fat, and the Detection of Cocoanut Oil in Butter Fat, 497
Pine Oil, in Turpentine Oil, Detection of, 143

Pipette, Automatic Pressure, 153
Pitch, Mineral Oils, etc., Asphalt in, Estimation of, 58
——, the Melting-Point of, New Method for Determining, 62
——, the Viscosity of, Comparison of Methods for Determining, 413
Plant Juices, the Oxidase Content of, Measurement of, 261
—— Substances, Inorganic Phosphorus in, Estimation of, 472
Plants, Small Quantities of Copper in, Detection and Estimation of, 313
——, the Amino-Acids in, Estimation of, by Means of the Formaldehyde Method, 312
Plates, Alundum, Filtration with, 109
Platinum, Action of Boiling Sulphuric Acid on, 590
———— Alloys, Analysis of, 420
————, Gold and Silver, Estimation of, 281
———— Salts, Action of Dimethyl-Glyoxime on, 524
————, Small Amounts of, in Ores and Bullion, Direct Determination of, 281
Wire, Use of Magnesia Rods instead of, in Analytical Work, 222
Poisonous Gases from Oilfields, 486
Polarimetric Estimation of Banana Starch, 560
———— ———— of Glucosamine in Ovomucoid and Pseudomucin, 561
———— ———— of Starch, 351
of Starch in Sausages, 498
Method, Lintner's, for the Determination of Starch, 498
Polarisation, Sodium Flame Lamps for, 594
Polenske and Kirschner Values for Margarines Containing Cocoanut or Palm-Kernel Oils, on the Relation between, 183
———— Value of Fats, the Estimation of, Sources of Error in, 256
Polypeptides and Some Amino-Acids, Hydrolytic Action of, on Certain Esters. Studies on Enzyme Action, 353
————, Mono-Amino-Acids together with, the Detection of, Ester Method for, 272
Portland Cement, Free Lime in, Use of Higher Phenols in Testing for, 582
Potash Analysis (Potassium-Sodium Cobaltinitrite Method), 588
Potassium in Potassium Silicate, Estimation of, 588
———— Iodide and Mercurous Nitrate as Sensitive Reagents for Tungsten and Molybdenum, 378
————, Much, the Estimation of Small Quantities of Rubidium in Presence of, Spectroscopic Process for, 324
———— Nitrate in Meats, Estimation of, 140
———— Permanganate, Estimation of Formic Acid in the Presence of Other Acids by Means of, 270
———————— Solution, Standardisation of, by Sodium Oxalate, 280
———— Silicate, Potassium in, Estimation of, 588
————, Small Quantities of, Estimation of, 214
---, Volumetric Estimation of, 34
Potatoes, Dry Matter in, Estimation of, and the Applicability of the Ordinary Tables, 259
Powder, Smokeless, Mercury in, Estimation of, 273
Powders, Cocoa, Husk in, Estimation of, 402
————, Nitrocellulose, and Nitrocellulose, Alteration of, by Heat, 104
————, Vegetable, the Estimation of Adulterants in, Apparatus for, and Notes on Starch Granules, 529
Precipitant for Amino-Acids, 359
Precipitates, Apparatus for Automatically Decanting and Washing, 479
————, Small Quantities of, the Quantitative Manipulation of, 68
Precipitation of the Copper-Arsenic Group and Separation of its Divisions, 376

Precipitin Reaction, the Quantitative, Application of, in Honey Analysis, II., 404
Preservative Coatings and Paint Films for Iron and Steel, New Method for Testing, 213
————, New, for Milk, Cream, etc., Note on, 178
Preservatives, Certain, Influence of, on the Keeping Properties and Composition of Butter, 52
———— for Milk, Cream, etc., Detection of Certain Proprietary Substances Sold as, Report to the Local Government Board on Analyses and Methods of. 155
————, Wood, the Commercial Value of, Tests to Determine, 578
Pressure, Great, the Course of Reactions under, Electric Furnace for Studying, 288
Primary and Secondary Amines, the Separation of Tertiary from, New Method for, 201
Producer Gas, Tar in, Estimation of, 275
Products, Natural, Sugars in, Estimation of, 145
————, Petroleum and its, Water in, Determination of, 573
Protein, Alcohol-Soluble, or Gliadin, in Wheat-Flour, Determination of, 19
Proteins, Certain, Refractive Indices of Solutions of. VI. The Proteins of Ox Serum: A New Optical Method for the Estimation of the Various Proteins in Ox Serum, 355
Proteins, Determination of the Power of Bacteria to Decompose, Method of Testing Water, 264
————, Estimation of Lactic Acid in the Presence of, 403
————, Other, and Horny Tissue, the Quantity of Tryptophane in, and Colorimetric Method for the Estimation of Tryptophane, 565
————, the Complete Hydrolysis of, Conditions for, 573
Proteolytic Enzyme of Malt, 504
Proximate Analysis of Complex Lipoids by Alcoholysis, 463
Pseudomucin and Ovomucoid, Glucosamine in, Polarimetric Estimation of, 561
Public Health (Regulations as to Food) Act, 1907, Regulations under the, 432
Pulp, Sulphite, Bleaching Quality of, Determination of, 511
Pumps, Filter, Valve for, 480
Purification, Softening, and Sterilisation of Water Supplies, 334
Pyridine and Ammonia, Separation and Estimation of, 413
———— Bases in Ammonia and Ammonium Salts, Detection of, 413
———— in the Presence of Ammonia in Gas-Water, Rapid Estimation of, 574
Pyrites and Sulphur, Selenium in, Iodimetric Estimation of, 215
Method for the Estimation of, 67
————, Copper in, Electro-Analytical Estimation of, 468
————, Sulphur in, Estimation of, 34
————, ————, Exact Determination of, 524
Pyroligneous Acid, Methylcyclopentenolon in, Occurrence of, 319
Pyrrolidonecarboxylic Acid, Separation of, from Glutamic Acid. Esterification of Monamino Acids by Means of Ethyl Iodide, 360

Qualitative Analysis, System of, for the Common Elements, Part V. Acidic Constituents, 322
———— without Hydrogen Sulphide, 377
———— Tests, Certain, for Oils, Influence of Lucidol (Benzoyl Peroxide) on, 273
Quantitative Analysis of Solutions by Precise Thermometry, New Method for, 333
Quinine in Urine and in Blood, Quantitative Estimation of, 455

Radium in Minerals and Rocks, Estimation of, 323, 421
————, the Comparison of Quantities of, New Compensation Method for, and Some Applications of this Method, 473

Rare Earths, Certain, Fusion of, with Alkali Carbonates. Separation of Tungsten from Iron, Beryllium, and Aluminium, 591

————, Separation of the, 380

Raw Materials, Cement, and Cement, Analysis of, 208

—— Rubber, the Amount of Insoluble Particles in, a Method for Determining, 13

Rays, Ultra-Violet, in Ozone, Absorption of, and its Application to the Estimation of Ozone in Small Concentrations, 472

——————, Sterilisation of Water and Dairy Materials by, 196

Reagent for Ferrous Salts, Dimethylglyoxime as a, 102

———, the, "Tetramethyl Base," Use of, 105

———, the Grignard, Quantitative Estimation of Hydroxy-, Amino-, and Imino-Derivatives of Organic Compounds by Means of, and the Nature of the Changes Taking Place in Solution, 201

Reagents, Sensitive, for Tungsten and Molybdenum, Potassium Iodide and Mercurous Nitrate as, 378

Red Rubber Goods, Antimony in, Estimation of, 64, 65

Reducing Agent, a New, for Analytical Use, Hydrazine Hydrate, and Formaldehyde, Tetraformal-trisazine from, 369

———— Sugar Methods (A Correction for Lactose), Unification of, 145

———— Sugars, Direct Determination of Sucrose in the Presence of, 451

——————, the Determination of the, New Method for, 205

——————, the Estimation of, by Fehling's Solution, Influence of Peptones on, 367

Reductase, Catalase, and Leucocyte Tests, Comparative Value of, in Detecting Mastitis, 401

———— Test, Acidity, and Alcohol Test in Milk, Relations between, 26

Redwood's, Engler's, and Saybolt's Viscosimeters, Comparative, Examination of, 223

Refractive Index of Beeswax, 348

—————— of Chinese Wood Oil, 410

——————, Standard, Rohrbach Solution of, Determination of the Density of Minerals by Means of, 148

———— Indices of Solutions of Certain Proteins. VI. The Proteins of Ox Serum: A New Optical Method for the Estimation of the Various Proteins in Ox Serum, 355

Refractometer Value of the Serum of Cow's and Goat's Milk, 317

—————, Zeiss', Estimation of Fat in Cocoa by Means of, 495

Refractometric Estimation of the Strength of Formaldehyde Solutions, 202

Rennet, Commercial, the Activity of, the Estimation of, 564

Rennin and Pepsin, Separation of, by Means of the Electric Current, 141

———, Laboratory Studies of, 405

(Report, Eighth, on Research Work. Metropolitan Water Board.) The Softening, Purification, and Sterilisation of Water Supplies, 334

———, Investigations of Explosives Used in Coal-Mines, 290

———, ————— of Fuse and Miner's Squibs, 291

———, Milk and Cream Regulations (Draft), 109

——— of the Government Chemist upon the Work of the Government Laboratory for the Year Ending March 31, 1912, with Appendices, 530

——— on the Work of Inspectors of Foods, 71

———, Seventh, on Research Work of the Metropolitan Water Board, 195

———, Sixth Annual, Metropolitan Water Board, 597

———, Study Committee of the International Dairy Federation, 532

———, The Connecticut Agricultural Experiment Station. Thirty-third and Thirty-fourth Reports, 1909 and 1910, 72

Report to the Local Government Board on Analyses and Methods of Detection of Certain Proprietary Substances Sold as Preservatives for Milk, Cream, etc., 155
——— to the Local Government Board on the Nature of the Colouring Matter of Flour and its Relation to Processes of Natural and Artificial Bleaching, 596
Resin, Scammony, and Scammony Root, Composition of, 194
"Resins, Acid," from Petroleum Residues, 515
Resins, Bitter, of Hops in Worts and Beers and their Volumetric Estimation, 139
———, Gum, Notes on the, 270
———, the Amount of, and the Viscosity of Rubber Solutions, Relationship between, 575
———, Total Soft, and Hard, in the Hop, Determination of, Comparative Study of Methods for, 191
Riegler's Method, Estimation of Urea in Urine by. A Correction Factor, 357
——————— for the Estimation of Urea in Urine, Modification of, 100
Ritter Method, the Estimation of Cholesterol by the Errors in; Effect of Autolysis on Cholesterol, 199
Roasted Blende, Sulphur in, Rapid Method for Determination of, 150
Rocks and Minerals, Radium in, Estimation of, 323, 421
———, Silicate, Ferrous Iron in, Estimation of, 146
Rods, Magnesia, Use of, instead of Platinum Wire, in Analytical Work, 222
Rohrbach Solution of Standard Refractive Index, Determination of the Density of Minerals by Means of, 148
Rosin and Mineral Oils, Fluorescence Test for, 30
Rubber, Direct Determination of, 574
——— Goods, Red, Antimony in, Estimation of, 64, 65
———, Raw, the Amount of Insoluble Particles in, a Method for Determining, 13
——— Solutions, the Viscosity of, and the Amount of Resins, Relationship between, 575
———, the Estimation of, Hübener Bromide Method for, 465
Rubberised Fabrics, Analysis and Testing of, 575
Rubidium, Small Quantities of, the Estimation of, in Presence of Much Potassium, Spectroscopic Process for, 324
Rum, Guadaloupe, Composition of, 97
Rye, Ergot of, a New Glucoside in: Clavicepsin, 19

Saccharin and Dulcin in Beverages, Foods, and Drugs, Separation and Identification of, 23
———— and Saponin in Oil Emulsions, Estimation of, 22
———— in Foods, Estimation of, 194
Saffron, Adulterants in, Estimation of, 309
Sale of Food and Drugs Acts, 1875 to 1907, Circular to Local Authorities for the Purposes of, 434
Salicylates, Thymol, and Allied Compounds, Estimation of, 369
Salicylic Acid and Copper, Colorimetric Estimation of, 104
——————, Small Quantities of, in Presence of Excess of *p*-Hydroxybenzoic Acid, Biochemical Method for the Estimation of, 316
Salt, a Neutral, and Alcohol, Simultaneous Effect of, on the Sensitiveness of Phenolphthaleïn, 321
—— Solution, Normal, Solubility of Ether in, 448
Salts, Ammonium, and Ammonia, Pyridine Bases in, Detection of, 413
——, Copper, Titration of, with Titanous Chloride, 516
——, Ferric, Compounds of, with Albumoses, 408

Salts, Ferrous, Dimethylglyoxime as a Reagent for, 102
——, Gold, Reaction of, with *m*-Phenylenediamine, 469
——, Lead, of the Higher Fatty Acids of Ether and in Petroleum, Ether, Solubilities of, 399
——, Platinum, Action of Dimethylglyoxime on, 524
——, the Penetration of, in Treated Wood, Determination of, 150
Sand, Monazite, Thorium, Especially in, Quantitative Estimation of, by Means of Sodium Hypophosphates, 423
"Sapometer," 479
Saponification of Fats by Sulphuric Acid, 256
——————, Fractional, 558
Saponin and Glycyrrhizin in Lemonades, Detection of, by Means of Vamvakas' and Frehse's Tests, 22
——— and Saccharin in Oil Emulsions, Estimation of, 22
———, Detection of, 350
———, in Beverages and Foods, Detection of, by Hæmolysis, 350
Sausages, Starch in, Polarimetric Estimation of, 498
Savin Oil, Detection of, in Toxicological Cases, 357
——, Oil of, the Constituents of, 295
Sawdust, Ordinary and Treated, the Value of, for Animal Nutrition, Experiments on, 559
Saybolt's, Engler's, and Redwood's Viscosimeters, Comparative Examination of, 223
Scale Preparations (Pharmaceutical), Iron in Determination of, 306
Scammony Resin and Scammony Root, Composition of, 194
Schlœsing's Method, Estimation of Nitrates and Nitrites in Water by, 212
Sea-Water, Concentration of Hydrogen Ions in, Measurement of, 425
Secondary and Primary Amines, the Separation of Tertiary from, New Method for, 201
Secretory Capacities, Amyloclastic, of the Embryo and Aleurone Layer of Hordeum, 97
Seeds of *Illicium Verum* Hook and *Illicium Religiosum* Sieb, the Fatty Oils obtained from, Composition of, 495
Selenium in Sulphur and Pyrites, Iodimetric Estimation of, 215
——————————, the Estimation of, Method for, 67
Seliwanoff Lævulose Reaction, Physico-Chemical Basis of, 367
Sensitive Reaction of Free Bromine, 581
——— Reagents for Tungsten and Molybdenum, Potassium Iodide and Mercurous Nitrate as, 378
——— Test for the Detection of Albumin in Urine, 505
Sensitiveness of Phenolphthaleïn, Simultaneous Effect of Alcohol and a Neutral Salt on, 321
Serger's Reaction for Vegetable Oils, Notes on, 412
Serum of Cow's and Goat's Milk, Refractometer Value of, 317
——, Ox, the Proteins of: A New Optical Method for the Estimation of the Various Proteins in Ox Serum. Refractive Indices of Solutions of Certain Proteins, 355
Serums, Milk, Preparation of, and their Use in the Analysis of Milk, 450
Sesame Oil in Other Oils, Reaction for Detecting, 62
Sewage Effluents, Nitrates in, Estimation of, 149
Shea-Nuts and Shea-Butter, 496
Shellacs and Stick-Lacs, Various, the Examination of, Preliminary Notes on, 204
Shimer Crucible, the Newest Form of, Determination of Carbon in Steel by Direct Combustion in, with the Aid of a Perforated Clay Disc, 521
Shrewsbury and Knapp's Process for Estimating Cocoanut Oil, Notes on, 3
Siderurgical Products, the Rapid Estimation of Sulphur in, Modification of the Method of Arnold and Hardy for, 283

Silicate, Potassium, Estimation of Potassium in, 588
——— Rocks, Ferrous Iron in, Estimation of, 146
Silicates, Alkalis in, Estimation of, by Fusion with Calcium Chloride, 146
———, the Estimation of Water in, Improvements in the Ludwig-Sipöcz Method for, 380
Silicic Acid, Colloidal, Phosphoric Acid in Presence of, Estimation of, 214
——— Gels, Vapour Pressure Isothermals of, Apparatus for Determination of, 328
Silicomolybdates, Separation of Phosphomolybdates from, 107
Silver, Gold, and Platinum, Estimation of, 281
——— Spiral, New, for Use in Organic Elementary Analysis, 289
Slide, Electrically-Heated Microscope, 592
Smokeless Powder, Mercury in, Estimation of, 273
Soap, Water in, Estimation of, 31
SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS, PROCEEDINGS OF, 3, 41, 81, 121, 168, 229, 295, 339, 392, 439, 485, 537
Soda Lime Glasses, the Refractive Index of, Relation of, to their Chemical Composition, 278
——, Nitrate of, Rational Analysis of, 523
Sodium Flame Lamps for Polarisation, 594
——— Hydroxide and Sugar, Hydrogen Peroxide in Presence of, Quantitative Separation of Copper from Arsenic, Aluminium, Zinc, Tungsten, and Tin by Means of, 210
——— Hypophosphate, Quantitative Estimation of Thorium, especially in Monazite Sand, by Means of, 423
——— Oxalate, Standardisation of Potassium Permanganate Solution by, 280
——— Peroxide for Bleaching Purposes, Assay of, 476
Softening, Purification, and Sterilisation of Water Supplies, 334
Soil Analysis, Mechanical, Modification of the Method of, 589
——, Hawaiian, Humus in, Determination of, 513
—— Humus, Studies on, 466
—— Phosphates, the Solubility of, Effect of Ignition on, 523
Soils, Arable, Colloids in, Estimation of, 98
——, Arid, Mechanical Analysis of, 588
——, Calcium Carbonate in, a Method of Estimating, 128
——, Phosphoric Acid in, the Phosphomolybdate Estimation of, 130
——, the Acidity of, Study of, 466
——, ————, —— on, 422
——, the Physical Analysis of, New Method for, 36
——, Total Manganese in, Estimation of, 423
Solanidine from *Solanum Tuberosum*, 497
Solanum Tuberosum, Solanidine from, 497
Solidified (Hydrogenised) Oils, 452
Solids and Liquids, Quantitative Separation and Estimation of, Laboratory Centrifuge for, 286
———, Density of, Determination of, New Densivolumeter for, 328
———, Total, of Vinegar, Estimation of, 260
———, ———, of Wine, the Direct Estimation of, Critical Remarks on, 311
Solubility Estimations in Agricultural Chemistry, 37
Solutions, Quantitative Analysis of, by Precise Thermometry, New Method for, 333
Solvates in Mixtures of Liquids, the Detection of, and Mixtures of Associated with Non-Associated Liquids (Part III). Latent Heats of Vaporisation of Mixed Liquids, 362

Solvents, Cold Estimation of Fat by Extraction with, 102
———, Immiscible, Apparatus for Extracting Liquids with, and for Measurement of Evolved Gases, 69
———, Non-Aqueous, Rare Earth Reactions in, 524
Soot, Its Character and Composition, 63
Souring of Milk, 168
Southern Cypress, Oil of the, 364
Soxhlet Extraction Apparatus, Modified, with Distillation Arrangement, 596
Sparking, Rapid Removal of Nitrogen from Gas Mixtures by, Apparatus for, 224
Specific Gravity of Some Fatty Oils, Effect of Free Fatty Acid on, 410
Spectra, Blood, the Intensity Distribution in, Photographic Estimation of, 313
Spectroscopic Process for the Estimation of Small Quantities of Rubidium in Presence of Much Potassium, 324
Spelter, Technical Analysis of, 521
Spermatic Fluid, Microchemical Reaction of, 358
Spices, etc., Small Quantities of Essential Oils in, the Estimation of, Part II., 88
Spiral, New Silver, for Use in Organic Elementary Analysis, 289
Spirits and Ethyl Alcohol, Methyl Alcohol in, Detection of, 567
———, Estimation of Methyl Alcohol Especially in, and in Admixture with Methyl Alcohol, 514
Springs, Thermal, of Bath, Analysis of the Waters of, 478
Squibs, Miners', and Fuse, Investigations of, 291
Stability Test, Modified Form of, 573
———, New, for Nitrocellulose Powders, 572
Standard, an Acidimetric, Benzoic Acid as, 458
Standardisation, Electrolytic, of the Conditions of the Iodide Method of Copper Analysis, and Sources of Error, 277
——— of Potassium Permanganate Solution by Sodium Oxalate, 280
Standards for Malt Vinegar, 123
———, Widely Varying, Rapid Work with, Colorimeter for, 329
Starch, Banana, Polarimetric Estimation of, 560
——— Granules, Notes on, and Apparatus for the Estimation of Adulterants in Vegetable Powders, 529
———, Indigos Containing, Analysis of, 319
———, in Sausages, Polarimetric Estimation of, 498
———, Lintner's Polarimetric Method for the Determination of, 498
———, Polarimetric Estimation of, 351
Starches, Commercial, for Use in Cotton Mills, Method for Determining the Value of, 368
Statistics, Fruit-Juice, for the Year 1911, 94
———, Wine, for the Year 1909-10, 96
Statutory Rules and Orders, 435
Steel and Iron, Oxygen in, Determination of, by Reduction in an Electric Vacuum Furnace, 586
———, Paint Films and Preservative Coatings for, New Method for Testing, 213
———, Sulphur in, Volumetric Estimation of, 67
———, Carbon in, Determination of, by Direct Combustion in the Newest Form of Shimer Crucible, with the Aid of a Perforated Clay Disc, 521
the Estimation of, 392
———, Determination of Vanadium in, New Method for, 285
———, Vanadium in, Rapid Determination of, 283
Steels, Ores, etc., Rapid Method for, Determination of Vanadium in, Based on its Quantitative Inclusion by the Phosphomolybdate Precipitate, 284

Steels, Vanadium, Estimation of Chromium and its Separation from, 146
Sterilisation of Water and Dairy Materials by Ultra-Violet Rays, 196
———, Softening, and Purification of Water Supplies, 334
Stick-Lacs, and Shellacs, Various, the Examination of, Preliminary Notes on, 204
Storax, Examination of, 499
Sublimation Apparatus, New, and its Application in Food Analysis, 428
——— in a Vacuum, Simple Apparatus for, 38
——— Vacuum, New Apparatus for, 290
Succinic and Malic Acids in Wines, Estimation of, 453
Sucrose in Condensed Milk, Estimation of, 450
——— in the Presence of Reducing Sugars, Direct Determination of, 451
———, Inversion of, by the Enzymes of Honey, 194
——— Solutions, Inversion of, by Means of Ammonium Chloride, 576
Sugar Analysis by Copper Reduction Methods, a Critical Study of, 413
——— and Sodium Hydroxide, Hydrogen Peroxide in Presence of, Quantitative Separation of Copper from Arsenic, Aluminium, Zinc, Tungsten, and Tin by Means of, 210
——— Determinations, Invert, Electrolytic Apparatus for Use in, 576
——— in Blood, Estimation of, 141, 263
——— in Urine and Blood, Estimation of, 356
———, the Estimation of, Use of Charcoal as a Clarifying Agent in, 57
———, Trommer's Test for, 406
———, Invert, Expanded Meissl-Hiller Table for, 575
———, ———, in the Presence of Alkali and Hydrogen Peroxide, Behaviour of, 28
———, Reducing, Methods (A Correction for Lactose), Unification of, 145
Sugars and Alcohol, the Iodimetric Titration of Sulphites in Presence of, 439
——— in Natural Products, Estimation of, 145
———, Other, Estimation of Cane-Sugar in Urine in the Presence of, 506
———, Reducing, Direct Determination of Sucrose in the Presence of, 451
———, ———, the Determination of the, New Method for, 205
———, ———, the Estimation of, by Fehling's Solution, Influence of Peptones on, 367
———, the Estimation of, New Process for, 63
Sulphates in Solution, Estimation of, by the Physico-Chemical Volumetric Method, 282
——— in Water, the Determination of, Modification of the Modified Winkler Method for, 220
Sulphides in Lime Liquors, Estimation of, 325
———, Insoluble, Sulphur in, Estimation of, 283
Sulphite Pulp, Bleaching Quality of, Determination of, 511
Sulphites in Presence of Alcohol and Sugars, the Iodimetric Titration of, 439
Sulphonation Test for Creosote, Modification of, 269
Sulphur and Halogens in Small Quantities of Organic Substances, Estimation of, 216
——— and Pyrites, Selenium in, Iodimetric Estimation of, 215
———, ———, Method for the Estimation of, 67
——— Compounds in Commercial Benzol, Estimation and Elimination of, 100
———, Free, Estimation of, Rapid Volumetric Method for, 151
——— in Illuminating Gas, Estimation of, 152
——— in Insoluble Sulphides, Estimation of, 283
——— in Iron and Steel, Volumetric Estimation of, 67
——— in Nitrocellulose, Estimation of, 152, 273
——— in Petroleum, Estimation of, 217
——— in Pyrites, Estimation of, 34
———, Exact Determination of, 524
——— in Roasted Blende, Determination of, Rapid Method for, 150

Sulphur in Siderurgical Products, the Method of Arnold and Hardy for the Rapid Estimation of, Modification of, 283
——— in Steel and Iron, Volumetric Estimation of, 67
Sulphuric Acid, Boiling, Action of, on Platinum, 590
————, Estimation of, 152
————, Nitric Acid, and Nitrous Acid, in Nitrating and Waste Acids, Volumetric Estimation of, 381
————, Rapid Method of Purifying, 381
————, Saponification of Fats by, 256
———— Solution, the Method of Precipitating Metals as Iodides, etc., from, Applications of, 519
Sweeney Method for Crude Fibre, Modification of the, 447
Sweetened Condensed Milk, Australian, Composition of, 49
Syrups, Formic Acid in, Detection and Estimation of, 558
——, Gum in, Estimation of, 94
——, Process of Extracting Alkaloids from, 138

Tablets, Medicinal, Lecithin in, Estimation of, 449
Tannin in Wines, Estimation of, 195
Tannins, Vegetable, Detection and Differentiation of, 577
———, ———, Quantitative Reactions of, 31
Tar in Producer Gas, Estimation of, 275
Tartaric Acid, the Estimation of, General Method for, 516
Tea-Seed Oil and Cake, 500
Temperatures, Low (−36° to −83° C.), Ebullioscopic Determinations at, 287
Tertiary Alcohols, Detection of, 197
——— Amines, the Separation of, from Secondary and Primary Amines, New Method for, 201
Tetrabromide, Estimation of Caoutchouc as, 361
Tetraformal-trisazine, from Formaldehyde and Hydrazine Hydrate, a New Reducing Agent for Analytical Use, 369
"Tetramethyl Base," the Reagent, Use of, 105
Textile Fibres and Woods, Cellulose in, Estimation of, 268, 510
——— Finishes, Analysis of, 200
Thermometer, Beckmann, with a Modified Arrangement for Setting the Mercury Thread, 429
Thermometry, Precise, Quantitative Analysis of Solutions by, New Method for, 333
Thermo-Regulators, 108
Thermostat, Vapour, Easily Adjustable, 430
Thermostats, Shaking and Stirring Apparatus, etc., Notes on, 70
Thimble, Extraction, New Form of, 288
Thorium, New Reagent for, 382
———, Quantitative Estimation of, Especially in Monazite Sand, by Means of Sodium Hypophosphate, 423
———, the Separation of, New Method for, 219
Thymol, Salicylates, and Allied Compounds, Estimation of, 369
Thyroid Glands, Iodine Content of, 456
Timber, Creosote Oil and Other Oils Used for Preserving, Antiseptic Effect of, 56
Tin and Antimony, Bronzes Containing, Chromium in, Estimation of, 374
——, Arsenic, Aluminium, Zinc, and Tungsten, Quantitative Separation of Copper from, by Means of Hydrogen Peroxide in Presence of Sodium Hydroxide and Sugar, 210
——, Copper, Antimony, and Bismuth, Electro-Analysis of, with Acidified Chloride Constituents, 584

Tin, Volumetric Determination of, 477
Tissue, Horny, and Other Proteins, the Quantity of Tryptophane in, and Colorimetric Method for the Estimation of Tryptophane, 565
Tissues, Animal, the Drying of, Loss of Fat during, 355
———, Living, Waters, and Minerals, Minute Quantities of Fluorine in, Detection and Estimation of, 517
Titanium, New Colorimetric Method for, 590
————, Separation of Iron from, 323, 469
————, Volumetric Determination of, 590
Titanous Chloride, Titration of Copper Salts with, 516
Tobacco Extracts, Nicotine in, Methods of Estimating, 452
———, Nicotine in, Estimation of, 311
Tobaccos, Ceylon, Analyses of, 500
Toxicity of Mushrooms: Their Hæmolytic Action, 563
Toxicological Cases, Detection of Savin Oil in, 357
Traces of Arsenic and Antimony, Detection of, 581
Tragacanth, Gum, Volatile Acidity of, Compared with that of Indian Gum, 318
Trommer's Test for Sugar in Urine, 406
Tryptic Activity, Relative, Measurement of, 457
——— Value of Pancreatin, New Method for Determining, 26
Tryptophane, the Estimation of, Colorimetric Method for, and the Quantity of Tryptophane in Horny Tissue and other Proteins, 565
Tube, Modified Butyrometer, for the Estimation of Fat in Cheese, 221
Tungsten Alloys, High-Grade, Analysis of, 424
———— and Molybdenum, Sensitive Reagents for, Potassium Iodide and Mercurous Nitrate as, 378
————, Arsenic, Aluminium, Zinc, and Tin, Quantitative Separation of Copper from, by Means of Hydrogen Peroxide, in Presence of Sodium Hydroxide and Sugar, 210
———— in Ferrotungsten, Rapid and Exact Estimation of, 220
————, Separation of, from Iron, Beryllium, and Aluminium. Fusion of Certain Rare Earths with Alkali Carbonates, 591
Turmeric and Aniline Dyes in Mustard, Detection of, 557
Turpentine, Examination and Valuation of, 370
—————, Other Hydrocarbons in, Detection of, 311
—————, Oil of, the Purity of, Tests for Determining, 371
————— -Petroleum Spirit and Benzene Hydrocarbons in, Estimation of, 274
—————, Pine Oil in, Detection of, 143
—————, Petroleum in, Estimation of, 371

Ultrafiltration in Analytical Chemistry, Application of, 223
Ultra-Microscope, Value of Direct enumeration of Bacteria in Water by Means of the, 317
———-Violet Rays in Ozone, Absorption of, and its Application to the Estimation of Ozone in Small Concentrations, 472
————————, Sterilisation of Water and Dairy Materials by, 196
Uranyl Solutions, Determination of Phosphorus in Food-Stuffs and Other Organic Substances by Titration with, 258
Urea, Estimation of, 27
—— in Urine, Estimation of, by Riegler's Method. A Correction Factor, 357
——————, Riegler's Method for the Estimation of, Modification of, 100
Uric Acid and Other Substances, Blue Colour-Reaction of Phosphotungstic Acid with, 355
—————, Colorimetric Method for the Estimation of, 358

Uric Acid Reaction, New, 276
Urine, Albumin in, the Detection of, Sensitive Test for, 505
——, Albumoses in, Detection of, 24
—— and Blood, Dextrose in, Estimation of, 140
————————, l-β-Hydroxybutyric Acid in, Quantitative Estimation of, 314
————————, Quinine in, Quantitative Estimation of, 455
————————, Sugar in, Estimation of, 356
——, and Other Physiological Fluids, Blood in, Detection of, 506
——, Benzoic Acid, Hippuric Acid, and Phenaceturic Acid in, Estimation of, 356
——, Cane-Sugar in, Estimation of, in the Presence of Other Sugars, 506
——, Diabetic, Glycuronic Acid in, Detection of, 507
——, Hippuric Acid in, the Estimation of, Method for, 507
——, ———— ——, New Method for, 356
——, Normal, Combined, Glycuronic Acid in, Detection of, 562
——, ————, Copper-Reducing Power of, 264
——, Phenol in, Estimation of, 356
——, Sugar in, the Estimation of, Use of Charcoal as a Clarifying Agent in, 57
——, ————, Trommer's Test for, 406
——, the Acidity of, Estimation of, 141
——, Urea in, Estimation of, by Riegler's Method. A Correction Factor, 357
——, ————, Riegler's Method for the Estimation of, Modication of, 100
——, Urobilinogen of, and Ehrlich's Reaction for Aldehydes, 28
Urobilinogen of Urine and Ehrlich's Reaction for Aldehydes, 28
Uva-Ursi Leaves, the Analysis of, Application of the Biochemical Method to, 24

Vacuum Furnace, an Electric, Determination of Oxygen in Iron and Steel by Reduction in, 586
——, Sublimation in a, Simple Apparatus for, 38
——, ————, New Apparatus for, 290
Value, Iodine, and the Structure of Acids of the Oleic Series, Relationship between, 463
——, the Acetyl Estimation of, 508
"Value," the "Gasolene Colour," of Flour, the Determination of, Method for, 255
Valve for Filter Pumps, 480
Vamvakas' and Frehse's Tests, Detection of Saponin and Glycyrrhizin in Lemonades by Means of, 22
Vanadium, Determination of, New Method for, 286
———— in Steel, Determination of, New Method for, 285
———— ————, Rapid Determination of, 283
———— in Steels, Ores, etc., Determination of, Rapid Method for, Based on its Quantitative Inclusion by the Phosphomolybdate Precipitate, 284
———— Steels, Estimation of Chromium and its Separation from, 146
Vanillin in Flavouring Extracts, the Determination of, New Colorimetric Method for, 501
Vaporisation of Mixed Liquids, Latent Heats of. Part III: Mixtures of, Associated with Non-Associated Liquids, and the Detection of Solvates in Mixtures of Liquids, 362
Vapour Pressure Isothermals of Silicic Acid Gels, Apparatus for Determination of, 328
———— Thermostat, Easily Adjustable, 430
Vapours, Chloroform, and Ether in Air, Gasometric Estimation of, 513
Vegetable Fats, Some, Analytical Constants of, 349
———— Foods, Arsenic in, Amounts of, 254
———— Oils and Fats, Certain, Bromine Absorption of, 403

Vegetable Oils and Paints, Heavy Petroleum in, the Detection of, 247
Serger's Reaction for, Notes on, 412
Powders, the Estimation of Adulterants in, Apparatus for, and Notes on Starch Granules, 529
Substances, Oxalic Acid in, in Estimation of, 564
Tannins, Detection and Differentiation of, 577
Qualitative Reactions of, 31
Vinegar, Distilled, when Stored in Wooden Casks, the Acid-Content of, Alteration of, 23
———, Malt, Standards for, 123
Viscosimeters, Engler's, Redwood's, and Saybolt's, Comparative Examination of, 223
Viscosity, Absolute, Method of Measuring, 154
——— of Fish Oils, 143
——— of Pitch, Methods for Determining, Comparison of, 413
——— of Rubber Solutions and the Amount of Resins, Relationship between, 575
Volatile Acidity of Gum Tragacanth Compared with that of Indian Gum, 318
——— Liquids, Molecular Weights of, Determination of, 595
——— Oils, Cineol in, Estimation of, 461
——— Organic Liquids, Combustion of, 361
——— Substances, a Device for the Combustion of, Estimation of Halogens in Organic Compounds, Including, 461
——— ——— in Coal, the Estimation of, Influence of Metallic Carbonates on, 360
Volatility of Arsenious Chloride, 805
Von Lorenz's Method, Estimation of Phosphoric Acid by, 149

Water, Aerated, the Bacteriology of, 395
——— Analysis, Geochemical Interpretation of, 107
——— and Dairy Materials, Sterilisation of, by Ultra-Violet Rays, 196
———, Apparatus for Testing, by Measurement of its Electrical Conductivity, 538
———, Bacteria in, Direct Enumeration of, by Means of the Ultra-Microscope, Value of, 317
——— Board, Metropolitan Sixth Annual Report, 597
———, Continuous Fractional Distillation of, 474
———, Free Carbon Dioxide in, Estimation of, by Titration with Alkalis in the Presence of Phenolphthaleïn, 582
——— in Butter, Estimation of, 51
——— in Molasses, Determination of, 560
——— in Petroleum and its Products, Determination of, 560
——— in Silicates, the Estimation of, Improvements in the Ludwig-Sipöcz Method for, 380
——— in Soap, Estimation of, 31
——— "Luminator" Treatment of, 154
———, Method of Testing. Determination of the Power of Bacteria to Decompose Proteins, 264
———, Nitrates and Nitrites in, Estimation of, by Schlœsing's Method, 212
———, Sea, Concentration of Hydrogen Ions in, Measurement of, 425
———, Sulphates in, the Determination of, Modification of the Modified Winkler Method for, 220
Supplies, the Softening, Purification, and Sterilisation of. (Metropolitan Water Board. Eighth Report on Research Work), 334
Waters, Carbonated, Ammonia in, the Estimation of, 173
——— of the Thermal Springs of Bath, Analysis of, 478

Waters, Minerals, and Living Tissues, Minute Quantities of Fluorine in, Detection and Estimation of, 517
Wax, Carnauba, and Beeswax, Hydrocarbons in, Estimation of, 348
——, Cotton, Characteristics of, 203
——, Paraffin, and Ceresin, Mixtures of, Analysis of, 59
——, ———, the Analysis of, Application of the Formolite Reaction to, 274
Wheat-Flour, Gliadin or Alcohol-Soluble Protein in, Determination of, 19
Wine, Glycerol in, Estimation of, 260
——, Malic and Succinic Acids in, Estimation of, 453
——, Phosphorus in, Estimation of, 24
——, Small Quantities of Zinc in, Detection of, 195
——, Statistics for the Year 1909-1910, 96
——, Succinic and Malic Acids in, Estimation of, 453
——, the Total Solids of, the Direct Estimation of, Critical Remarks on, 311
Wines from Eastern France, Analyses of, 352
——— from the Department of Gironde, Analyses of, 352
——, Phosphoric Acid in, Estimation of, 453
——, Tannin in, Estimation of, 195
Winkler Method, the Modified, Modification of, for the Determination of Sulphates in Water, 220
Wohlgemuth's Method for the Quantitative Investigation of the Pancreas Function, Modification of, 315
Wood Oil, Chinese, Heat Test for, 410
————, ————, Method of Examining, 410
————, ————, Refractive Index of, 410
————, ———— and Japanese, the Examination of, 543
—— Preservatives, the Commercial Value of, Tests to Determine, 578
——, Treated, the Penetration of Salts in, Determination of, 150
Woods and Textile Fibres, Cellulose in, Estimation of, 268, 510
Wort and Beer, Bitter Principles in, Estimation of, 92
————————, Extract in, Estimation of, 502
Worts and Beers, Bitter Resins of Hops in, and their Volumetric Estimation, 139

Xanthogenate, Arsenic, and its Use in Analysis, 569

Yttrium, Quantitative Determination of, 382
————, ——————— Separation of Lanthanum from, 469

Zeiss Interferometer in the Technical Analysis of Flue-Gases, Use of, 427
Zeiss' Refractometer, Estimation of Fat in Cocoa by Means of, 495
Zinc and Nickel, the Gravimetric Separation of, 443
——, Arsenic, Aluminium, Tungsten, and Tin, Quantitative Separation of Copper from, by Means of Hydrogen Peroxide in Presence of Sodium Hydroxide and Sugar, 210
—— in Ferruginous Minerals, Estimation of, 425
——, Rapid Method of Estimating, 31, 153
——, Small Quantities of, in Wine, Detection of, 195
——, the Ferrocyanide Titration of, Influence of Lead on, 585
Zirconium, Separation of, from Iron and Aluminium, and a New Method for the Analysis of Ferro-Zircon, 36

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THE ANALYST.

INTERNATIONAL ATOMIC WEIGHTS, 1912.

		O=16.
Aluminium	Al	27·1
Antimony	Sb	120·2
Argon	A	39·88
Arsenic	As	74·96
Barium	Ba	137·37
Bismuth	Bi	208·0
Boron	B	11·0
Bromine	Br	79·92
Cadmium	Cd	112·40
Cæsium	Cs	132·81
Calcium	Ca	40·07
Carbon	C	12·00
Cerium	Ce	140·25
Chlorine	Cl	35·46
Chromium	Cr	52·0
Cobalt	Co	58·97
Columbium	Cb	93·5
Copper	Cu	63·57
Dysprosium	Dy	162·5
Erbium	Er	167·7
Europium	Eu	152·0
Fluorine	F	19·0
Gadolinium	Gd	157·3
Gallium	Ga	69·9
Germanium	Ge	72·5
Glucinum	Gl	9·1
Gold	Au	197·2
Helium	He	3·99
Hydrogen	H	1·008
Indium	In	114·8
Iodine	I	126·92
Iridium	Ir	193·1
Iron	Fe	55·84
Krypton	Kr	82·92
Lanthanum	La	139·0
Lead	Pb	207·10
Lithium	Li	6·94
Lutecium	Lu	174·0
Magnesium	Mg	24·32
Manganese	Mn	54·93
Mercury	Hg	200·6
Molybdenum	Mo	96·0
Neodymium	Nd	144·3
Neon	Ne	20·2
Nickel	Ni	58·68
Niton (radium emanation)	Nt	222·4
Nitrogen	N	14·01
Osmium	Os	190·9
Oxygen	O	16·00
Palladium	Pd	106·7
Phosphorus	P	31·04
Platinum	Pt	195·2
Potassium	K	39·10
Praseodymium	Pr	140·6
Radium	Ra	226·4
Rhodium	Rh	102·9
Rubidium	Rb	85·45
Ruthenium	Ru	101·7
Samarium	Sa	150·4
Scandium	Sc	44·1
Selenium	Se	79·2
Silicon	Si	28·3
Silver	Ag	107·88
Sodium	Na	23·00
Strontium	Sr	87·63
Sulphur	S	32·07
Tantalum	Ta	181·5
Tellurium	Te	127·5
Terbium	Tb	159·2
Thallium	Tl	204·0
Thorium	Th	232·4
Thulium	Tm	168·5
Tin	Sn	119·0
Titanium	Ti	48·1
Tungsten	W	184·0
Uranium	U	238·5
Vanadium	V	51·0
Xenon	Xe	130·2
Ytterbium (Neoytterbium)	Yb	172·0
Yttrium	Yt	89·0
Zinc	Zn	65·37
Zirconium	Zr	90·6

OBITUARY.

PERCY GERALD SANFORD.

We regret to announce the sudden death, from apoplexy, on November 26, of Mr. Percy Gerald Sanford, F.I.C., at the age of fifty years. Mr. Sanford was a very old member of the Society of Public Analysts, and was Public Analyst for the Borough of Penzance and additional Public Analyst for the Borough of Croydon. He was educated at Dulwich College and at the Royal School of Mines, and was subsequently assistant for three years to the late Dr. C. Meymott Tidy at the London Hospital Medical College. He was also for a time research assistant to Professor Pickering at Bedford College. Mr. Sanford gave much attention to the chemistry of explosives, and was for a time resident chemist to the Stowmarket Explosives Company, and subsequently at the National Explosives Company's Works at Hayle, Cornwall. He also acted as consulting chemist to the Cotton Powder Company, Limited, and was the author of a well-known treatise on "Nitro-Explosives." During later years Mr. Sanford had been engaged in the practice of general analytical and consulting chemistry in London, carried on in conjunction with his work as a Public Analyst under the Sale of Food and Drugs Act. He leaves a widow and an infant daughter.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The monthly meeting of the Society was held on Wednesday evening, December 6, in the Chemical Society's Rooms, Burlington House. In the absence of the President the chair was occupied by Mr. Arthur R. Ling, F.I.C., Vice-President.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. S. G. Agar, G. S. Barton, S. W. Bridge, A.I.C., R. V. Briggs, F. A. Mason, W. B. Saville, G. A. Stokes, and F. C. Dyche-Teague, B.Sc., A.I.C., were read for the second time; and certificates in favour of Mr. Herbert Hawley, M.Sc. (Birmingham), City Analyst's Laboratory, Council House, Birmingham, assistant to Mr. J. F. Liverseege, F.I.C.; and Miss Maud Gazdar, B.Sc. (Lond.), A.I.C., Fox Hall, Upminster, Essex, assistant analyst to Messrs. Dakin Bros., manufacturing chemists, were read for the first time.

Messrs. R. D. Carty, W. K. Walton, and G. F. Wesley Martin, were elected members of the Society.

The following papers were read: "The Estimation of Small Quantities of Essential Oil in Spices, etc." (Part II.), by J. A. Brown, F.I.C.; "The Determination of Furfural by Means of Fehling's Solution," by Lewis Eynon, B.Sc., F.I.C., and J. Henry Lane, B.Sc., F.I.C.; "The Examination of Petroleum Mixtures," by J. H. Coste, F.I.C., E. T. Shelbourn, F.I.C., and E. R. Andrews, F.I.C.; "A Method for Determining the Amount of Insoluble Particles in Raw Rubber," by Clayton Beadle and Henry P. Stevens, M.A., Ph.D., F.I.C.; "Note on Oil of Male Fern," by Ernest J. Parry, B.Sc., F.I.C.; "Note on Ground Almonds," by G. Cecil Jones, A.C.G.I., F.I.C., and Reginald F. Easton; "Note on the Determination of Small Quantities of Methyl Alcohol," by C. Simmonds, B.Sc.; and "The Composition of Australian (Victoria) Milk," "The Composition of Sweetened Condensed Milk," and "The Aldehyde Figure of Butter," by E. Holl Miller.

The CHAIRMAN announced the Council's nominations of Officers and Council for 1912 as follows:

President.—L. Archbutt, F.I.C.

Past-Presidents (limited by the Society's Articles of Association to Eight in Number).—Edward J. Bevan, F.I.C.; Bernard Dyer, D.Sc., F.I.C.; Thomas Fairley, F.I.C.; W. W. Fisher, M.A., F.I.C.; Otto Hehner, F.I.C.; R. R. Tatlock, F.I.C.; E. W. Voelcker, A.R.S.M., F.I.C.; J. Augustus Voelcker, M.A., B.Sc., Ph.D., F.I.C.

Vice-Presidents.—W. H. Willcox, M.D., B.Sc., M.R.C.P., F.I.C.; Arthur R. Ling, F.I.C.; John E. Stead, F.R.S., F.I.C.

Hon. Treasurer.—H. Droop Richmond, F.I.C.

Hon. Secretaries.—A. Chaston Chapman, F.I.C.; P. A. Ellis Richards, F.I.C.

Other Members of Council.—E. Richards Bolton; R. M. Clark, B.Sc., F.I.C.; J. Connah, B.Sc., F.I.C.; J. Evans, F.I.C.; Charles A. Hill, B.Sc., F.I.C.; E. Hinks, B.Sc., F.I.C.; G. T. Holloway, A.R.C.Sc., F.I.C.; G. N. Huntly, B.Sc., A.R.C.Sc., F.I.C.; G. W. Monier-Williams, B.A., Ph.D., F.I.C.; F. Wallis Stoddart, F.I.C.; S. R. Trotman, M.A., F.I.C.; S. A. Woodhead, M.Sc., F.I.C.

* * * * *

NOTES ON SHREWSBURY AND KNAPP'S PROCESS FOR ESTIMATING COCOANUT OIL.

BY HERBERT S. SHREWSBURY, F.I.C., AND ARTHUR W. KNAPP, B.Sc., F.I.C.

(*Read at the Meeting, November* 1, 1911.)

MESSRS. CRIBB AND RICHARDS, in their paper on "Margarine Analysis" (ANALYST, 1911, **36**, 328), make the following statement: "Ample illustration of this is afforded by Shrewsbury and Knapp (ANALYST, 1910, **35**, 385) and other authors, who show that the differences between the figures for cocoanut oil and butter fat, upon which calculations are based, are not so great as in the Polenske process. The Polenske figure for cocoanut oil (17·5) is between five and six times that of butter (2 to 3·5), and, as far as we are aware, none of these other processes will improve on this ratio."

We cannot reconcile the first part of this statement with the figures actually given in our paper. The following table compares our method with the Polenske process in this respect. The figures are strictly comparable, being in both processes the number of c.c. $\frac{N}{10}$ alkali required for 5 grms. of fat:

	Figure for Cocoanut Oil.	Limit Figure for Genuine Butters.	Difference between Cocoanut Oil and Butter Figures.	Ratio of Cocoanut Oil and Limit Butter Figures.
Shrewsbury and Knapp...	163·0*	32·0*	131	5·1
Polenske	17·5†	3·5†	14	5·0

This comparison shows that the difference between the figures for cocoanut oil and butter fat is not *less* than that given by the Polenske method, but, on the contrary, more than nine times as great; and in practice we find that 5 per cent. of cocoanut oil increases the Polenske figure by about 0·5 c.c., and our figure by 5 c.c. This should be remembered when considering the very interesting figures found by H. T. Cranfield (ANALYST, 1911, **36**, 446). He showed that for butters from cows fed on cocoanut cake both the Polenske and our figure indicate the addition of cocoanut oil. Evidently the limit figure for genuine butters needs raising in both processes, to allow for the inclusion of these special butters amongst genuine butters. He showed, further, that genuine butters with a Reichert-Meissl value of 29·5 may give Polenske figures from 1·7 to 3·7—*i.e.*, a difference of 2. This difference has about the same significance as 20 in our process, for which the greatest variation found by Cranfield was 15·4 (40·1 – 24·7).

Again, Lewkowitsch found for edible cocoanut oils Polenske figures from 15·49 to 20·45. This wide range produces the same possible error in calculation as would a range for our process of 154·9 to 204·5. Happily, the range for cocoanut oils in our process is much less.

Whilst our method estimates the bulk of the fatty acids which occur as glycerides in cocoanut oil, the Polenske method depends upon the small percentage of water-insoluble volatile acids that are present. This difference is important, because it is not impossible that cocoanut oil could be treated to remove these, and thus give a Polenske figure approximating to that of butter. That the Polenske figure can be diminished by separating the liquid glycerides by pressure (as in the preparation of cocoa butter substitutes) is illustrated by the following analyses of products so obtained from a specimen of Cochin cocoanut oil:

	Reichert-Meissl Value.	Polenske Figure.	Shrewsbury and Knapp Figure.
Cocoanut "stearine" A... ...	4·2	12·1	169·0
Cocoanut "oleine" A	8·5	23·2	169·0
Cocoanut "stearine" B... ...	3·9	11·1	165·3

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* Ross, Race, and Maudsley (ANALYST, 1911, **36**, 196) found 16·55 to 17·05 for cocoanut oil, and 35·5 as maximum for foreign butters.

† Lewkowitsch found 15·49 to 20·45 for cocoanut oil, and 4·1 for a foreign butter.

(Cocoanut "stearine" B had been subjected to the well-known method of refining with superheated steam before being pressed.) Whilst the Polenske figure is greatly reduced by this treatment, the Shrewsbury and Knapp figure is not affected to any extent. That the figure for "stearine" A and "oleine" A should be identical is accidental, and indicates that the figure for the "oleine" has been *raised* by the large percentage of the glycerides of the lower fatty acids to just the same extent as it has been *lowered* by containing the greater part of the olein present in the original oil.

It has been pointed out that the Polenske method does not give such good results with palm-kernel oil, since its Polenske figure is only about 11. It is, in all probability, still less effective for estimating palm-kernel "stearine," which doubtless has a yet lower value. An old sample of palm-kernel "stearine" gave a Reichert value of 4·2, Polenske figure 9·35, and Shrewsbury and Knapp figure 162·9. It is noteworthy that the insoluble volatile acids were solid. Since the principal constituent of palm-kernel oil is laurin, the Shrewsbury and Knapp figure is of similar magnitude to the figure for cocoanut oil. A sample of crude palm-kernel oil gave 163·7.

As modifications of, or additions to, accepted processes are considered convenient, we made one or two experiments on the fatty acids left in the Reichert flask after the usual distillation of the fatty acids. The non-volatile acids from butter were treated as described by our method, but the figures obtained were abnormally high, and seem to indicate that the oleic acid undergoes some change whilst boiling with the sulphuric acid, glycerol, and sodium sulphate. The results were useless for our purpose.

Referring to the paper of Messrs. Revis and Bolton (ANALYST, 1911, **36**, 334), we cannot see the necessity or advantage of their proposed modifications of our process. Many of these modifications we tried and discarded; they greatly increase the time taken by the method, thereby rendering it unsuitable as a *sorting test*, without apparently increasing the accuracy. In our experience we found the tedious method of cooling, with the production of supersaturated solutions from which the fatty acids crystallise slowly, not so accurate as the rapid separation of the hot liquids. Another disadvantage of the modifications is that the range for pure butters, as the published figures show, is greater than in our process. Under one circumstance our process may require modification—that is, if it is required for giving accurate estimations where *large* percentages of cocoanut oil are present. In such cases the absolute control of the final conditions is of greater importance, and, for example, the use of a bath at 37° C. might possibly improve results. However, one of us, working in the tropics, obtained by our method normal butter figures and consistent duplicate results, although the laboratory temperature was 30° C. Evidently the method as it stands can be used under winter or summer conditions for the purpose for which it was intended—*i.e.*, the rapid detection and approximate estimation of small percentages of cocoanut oil.

	Figure for Cocoanut Oil.	Limit Figure for Genuine Butters.	Difference between Cocoanut Oil and Butter Figures.	Ratio of Cocoanut Oil and Limit Butter Figures.
Shrewsbury and Knapp...	163·0*	32·0*	131	5·1
Polenske	17·5†	3·5†	14	5·0

This comparison shows that the difference between the figures for cocoanut oil and butter fat is not *less* than that given by the Polenske method, but, on the contrary, more than nine times as great; and in practice we find that 5 per cent. of cocoanut oil increases the Polenske figure by about 0·5 c.c., and our figure by 5 c.c. This should be remembered when considering the very interesting figures found by H. T. Cranfield (ANALYST, 1911, **36**, 446). He showed that for butters from cows fed on cocoanut cake both the Polenske and our figure indicate the addition of cocoanut oil. Evidently the limit figure for genuine butters needs raising in both processes, to allow for the inclusion of these special butters amongst genuine butters. He showed, further, that genuine butters with a Reichert-Meissl value of 29·5 may give Polenske figures from 1·7 to 3·7—*i.e.*, a difference of 2. This difference has about the same significance as 20 in our process, for which the greatest variation found by Cranfield was 15·4 (40·1 – 24·7).

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Discussion.

Mr. C. Revis said that in his experience the Polenske figure could be determined with much greater accuracy than the Shrewsbury and Knapp figure. In the hands of Mr. Bolton and himself the Shrewsbury and Knapp process, as originally published, had not yielded concordant results. When the process was modified as Mr. Bolton and he had suggested, they found that quite concordant results could be obtained by different observers. The final cooling was very necessary in the case of mixtures containing large proportions of cocoanut oil, because the solubility of the final fatty acids in 60 per cent. alcohol depended greatly on the temperature.

Mr. T. Macara agreed with what Mr. Revis had said as to the difficulty of getting concordant results with the Shrewsbury and Knapp process when working at the higher temperature. In an ordinary laboratory it was not always easy to regulate the temperature exactly, and to be able to work at the ordinary temperature was a great advantage.

Mr. E. R. Bolton said that the experience of Mr. Revis and himself had been that even a slight variation in the time of running off the hot solution of fatty acids was liable to cause considerable differences in the results, and their object in modifying the process had been to enable more consistent results to be obtained by independent workers who merely follow a published description. That being accomplished the process would be found to be a valuable one. It was to be noted that many of the figures given in the author's present paper related to the whole fats—cocoanut "stearine," palm nut "stearine," etc. Mr. Revis and he, even by the Polenske method, had not obtained very satisfactory results when working on the whole undiluted fats, but with such admixtures as were generally found in margarine the results were delicate and accurate, duplicate Polenske figures obtained by different observers commonly agreeing within a tenth of a c.c., and as far as their experience went they considered that the ratio between the Reichert-Meissl and Polenske numbers, reinforced by the Kirschner figure, afforded the best means of estimating cocoanut oil now available (*cf.* Analyst, 1911, **36**, 333).

Mr. Knapp agreed that an essentially practical process, such as this, ought to be capable of being satisfactorily carried out, after a little practice, by an average worker who simply followed the published description. He thought, however, that some misconception had arisen as to the comparative accuracy of the two processes, owing to the fact that in this process the figures obtained were larger than in the Polenske process, so that an agreement within 0·1 c.c. in the Polenske process would correspond to an agreement within 1 c.c. in the Shrewsbury and Knapp process. For example, Mr. Cranfield had considered that duplicate results should agree within 0·5 c.c., and he (the speaker) concurred; but to reach a corresponding degree of accuracy in the Polenske process the figures would have to agree within 0·05 c.c. With regard to the question of time, the alcoholic solution of the fatty acids should be run off after three minutes, but it seemed hardly likely that, on one hundred and eighty seconds, a variation of one or two seconds either way would make any important difference.

Mr. Richards pointed out that in the paper referred to, Mr. Cribb and he had stated that none of the other processes improved materially on that of Polenske so far as the ratio employed for calculation was concerned, and the figures given by the present Authors confirmed this, as the difference between 5·0 and 5·1 is negligible.

* * * * *

NOTE ON A COUNTERFEIT GOLD COIN.

By HERBERT S. SHREWSBURY, F.I.C.

(Read at the Meeting, November 1, 1911.)

Analysts are not infrequently called upon to examine counterfeit coin; but the specimen described below, which was uttered in Trinidad, appeared to be so unusual in character as to be worthy of a brief description.

The sample imitated a twenty-shilling piece of date 1861. It weighed 122 grains against 123·27 grains for a genuine coin. The milling and design were very good, but the gilding had begun to wear off in places, and the partial exposure of the underlying white metal led to the detection of the false coin. A piece of the white metal, after removal of the gilding, gave a specific gravity of 21·8 $\left(\frac{30^{\circ}}{4}{}^{\circ}\text{ C.}\right)$ as against 17·157 for standard gold coinage, and had an approximate composition of:

Platinum	91·5	per cent.
Antimony and traces of bismuth and iron ...	5·0	,, ,,
Copper	3·5	,, ,,
	100·0	

It was quite infusible and unaltered in the hottest part of the flame of a Méker burner.

One or two notes on the analysis may be of interest. The metal was somewhat difficult of solution. 0·326 grm. of the white metal required more than three hours' treatment with aqua regia on the water-bath to effect its solution, and it was quite insoluble in nitric acid of any strength.

Insufficient notice seems to be taken in most textbooks of the difficulty of solution of platinum sulphide in ammonium sulphide. Fresenius is a notable exception, and points out that the sulphide precipitate requires prolonged treatment with a large excess of hot ammonium sulphide, and that even under these conditions some of the platinum sulphide is usually left undissolved.

A difficulty occurred in the qualitative detection of the copper. The presence of platinum in solution appears to prevent the formation of the characteristic blue coloration with ammonia. Some platinum sulphide was left undissolved with the copper sulphide after solution of the bulk of platinum sulphide and all the antimony sulphide in hot ammonium sulphide. This insoluble sulphide precipitate dissolved in strong nitric acid, forming a yellowish-green solution, but the colour of the solution was scarcely affected by the addition of excess of ammonia. Copper was at once detected, however, on treatment of the ignited ammonium platinum chloride precipitate with hot nitric acid and application of the usual tests to the green nitric acid extract of the reduced impure platinum.

Although the high specific gravity of platinum permits a counterfeit of this nature to be made of the correct weight, it is somewhat difficult to understand how any profits can accrue from the proceeding. The fact of such coins actually being in circulation is corroborated by a notice in the "Counterfeit Detector," in which bankers are warned of the existence of counterfeit sovereigns made of gilded platinum and dated 1862 and 1863.

Possibly at the time of their production the price of platinum was sufficiently low to allow the realisation of a large profit.

* * * * *

THE EXAMINATION OF MIXTURES CONTAINING PETROLEUM.

By J. H. COSTE, F.I.C., E. T. SHELBOURN, F.I.C., and
E. R. ANDREWS, F.I.C.

(*Read at the Meeting, December* 6, 1911.)

In Section 3 of the Petroleum Act, 1871, it is stated that "For the purposes of this Act the term 'petroleum' includes any rock oil, Rangoon oil, Burmah oil, oil made from petroleum, coal, schist, shale, peat or other bituminous substance, and any products of petroleum or any of the above-mentioned oils, and the term 'petroleum to which this Act applies' means such of the petroleum so defined as, when tested in manner set forth in Schedule One to this Act, gives off an inflammable vapour at a temperature of less than one hundred degrees of Fahrenheit's thermometer."

The manner prescribed was the unsatisfactory one of testing, by means of a small flame, the petroleum contained in an open cup heated in a water-bath.

Section 2 of the Petroleum Act, 1879, provided that the term "petroleum to which this Act applies" should in future mean petroleum which gave off an inflammable vapour at a temperature of less than 73° F. The apparatus then newly designed by the late Sir Frederick Abel was prescribed for use, and the temperature of flash 73° F., as registered by this apparatus, was intended to correspond to a flash of 100° F. in the open cup.

In 1899 the London County Council appealed against a magistrate's decision that a composition containing 20 per cent. of petroleum did not come within the meaning of the Acts, and the higher court remitted the case to the magistrate for him to convict. In 1907 the Privy Council, acting on a provision of the Act of 1871, endorsed this view of the law, and strengthened it by an Order "that certain parts of the 'Acts of 1871 to 1881' the latter being the petroleum (Hawkers) Act should apply to any mixture of petroleum with any other substance or substances which . . . gives off inflammable vapour at a temperature of less than 73 degrees of Fahrenheit's thermometer, whether such mixture be liquid, viscous, or solid, in the same manner as if such mixture were petroleum, to which the said Acts apply."

Dismissing from consideration several questions which may arise out of the wording of the above-mentioned Acts and Order, the points at issue for the analyst in connection with any mixture are (1) Does it contain petroleum as defined by the Acts? (2) Is it to be certified as petroleum to which the Acts apply?

It might be taken from the wording of part of the Order that any mixture in respect of which the answer to (1) was in the affirmative, and which gave off inflammable vapour below 73° F., was *ipso facto* within the scope of the Acts; but this is not necessarily so, as it is stated that "mixtures of petroleum with other substances present dangers similar to those presented by petroleum without admixture." It is therefore necessary to consider the second question from a somewhat wider point of view than the mere giving off of an inflammable vapour.

The fact that petroleum which may or may not flash below 73° F. is used in paints, enamels, paint removers, polishes, solvents, etc., in admixture with alcohol, ether, and acetone which flash below 73° F., and turpentines, carbon tetrachloride, trichlorethylene and tetrachlorethane, which do not, renders a decision as to the application of the Petroleum Acts one of greater difficulty than can be solved by the class of officer to whom testing under the Acts is often entrusted.

Since the Acts do not apply to mixtures flashing at or above 73° F., it is in all cases convenient to determine the flash-point first, leaving the decision as to whether samples which do flash are, or contain, petroleum within the meaning of the Acts, to be based on subsequent chemical work.

Apart from difficulties presented by certain liquids due to their containing chloro-compounds the determination of flash-point is not so simple or satisfactory as might be thought. The three well-known types of apparatus—the original Abel, the Abel-Pensky as used in the Colonies, and the German form of the same apparatus—have recently been very carefully examined and compared by J. H. Harker, and W. F. Higgins (*Nat. Phys. Lab. Coll. Researches*, VIII. 19-39). The following points have been studied by these workers: (1) The influence of variations in the method of testing (*a*) the frequency of application of the test-flame, (*b*) variations in the time of opening the slide, (*c*) variations in the water-bath temperature, (*d*) variations in the depth of immersion of the thermometer; (2) the temperature distribution in the oil-cup of the various forms of apparatus; (3) the heat from the test-flame, differences of ½° F. may occur even in the most careful work, more especially in the apparatus provided with the oil test-flame; (4) the difference between the flash-points determined with the different forms of apparatus. These are approximately constant at all temperatures; the German form gives results about 3·7° F. and the Colonial form 1·1° F. higher than the Abel. The oil test-flame reads about ½° F. higher than the gas-flame. (5) They emphasise the fact that the flash-point of a liquid, as ordinarily determined, is an empirical constant which is largely a function of the particular apparatus used. The effect of thermometric lag has also been investigated.

If a low flash-point establishes a *prima facie* presumption of the presence of petroleum to which the Acts apply, it is necessary, in the first place, to determine whether the flash is wholly or partly due to the presence of petroleum.

We outline a general scheme which should enable such a decision to be made. It is obvious that in many cases some of the processes suggested could be omitted or modified, and that at various stages much useful information will be given by the determination of such physical properties as refractive index, boiling-point, and specific gravity, and by the consideration of the relation of these constants to one another. The odour and the nature of the flash given by liquids will often furnish useful qualitative information.

A liquid, which on agitation with water is found to be wholly miscible with it, cannot contain any appreciable quantity of the hydrocarbons which are the main constituents of petroleum as known to the law. If any loss of volume occurs, owing to the solution of a portion of the sample, it should be washed with further quantities of water until all soluble constituents (alcohol, acetone, etc.) are removed. These washings, or the water coming over with the steam distillate, may be examined by determining the initial boiling-point of the liquid and the properties of the first portion of the distillate.

The following example will illustrate the ease with which this can be done: A Russian burning oil flashing at 81° F. was mixed with 10 per cent. of ether. After washing about 80 c.c. with 6 washings, in all 1850 c.c., the flash-point was found to be 82° F., showing that the tendency is rather for traces of petroleum to be lost than for ether to be retained.

Viscous liquids which obviously contain fixed oils or solids in solution or suspension can be distilled either alone at ordinary or reduced pressure or with steam in order to obtain a suitable liquid for working on. If this be done, it is necessary to ascertain that the residue contains no matters which were present originally in liquid petroleum—*e.g.*, a sample of ordinary burning oil, flashing at 84° F., was steam distilled, and gave 45 per cent. by volume of a distillate flashing at 69° F. An unwary worker examining a mixture which contained such an oil might be disposed to certify it as containing petroleum within the meaning of the Acts, whereas the process of fractionation was the cause of the low flash. In all doubtful cases it is advisable to wash repeatedly with water, then with sodium bisulphite solution to remove alcohols and acetone, and afterwards to dry with calcium chloride, which should remove any traces of alcohol, and again to take the flash-point (*cf.* Nash, ANALYST, 1911, **36**, 577).

Whether the sample has been distilled with steam or alone, or only washed with water, the treatment with sodium bisulphite and calcium chloride should be carried out on the sample or the distillate. Only hydrocarbons and their chlorine derivatives, amyl alcohol or acetate, and carbon disulphide, are now likely to be present. The hydrocarbons may be terpenes, paraffins and cycloparaffins, and aromatic hydrocarbons. Terpenes may be removed by Armstrong's method (*J. Soc. Chem. Ind.*, 1882, **1**, 480), which has been shown by one of us to be applicable to mixtures of terpenes with both aliphatic and aromatic hydrocarbons (Coste, ANALYST, 1910, **35**, 438; Coste and Nash, *ibid.*, 1911, **36**, 207). This treatment also removes the amyl compounds mentioned above, but in presence of these substances the acid must be removed before steam distillation, as they separate from solution when the acid is diluted. The remaining liquid can only contain paraffins and cycloparaffins, homologues of benzene, carbon disulphide, and chloro compounds. If 1 c.c. is poured into a small porcelain capsule, diluted with 2 c.c. of alcohol, and the capsule placed on the bottom of a large beaker covered with a clock glass, which is moistened on the under side with a drop of silver nitrate, the appearance of silver chloride on the drop when the diluted liquid is lighted and the clock glass placed over the beaker will indicate the presence of chlorine, and, by inference, of chloroparaffins, etc. It is seldom that this treatment is necessary, as a peculiar reddish, smoky flash and an odour of hydrochloric acid will usually indicate the presence of any of these bodies. Carbon disulphide is seldom found in admixture with other solvents, and does not need much consideration.

Veley (*Proc. Roy. Soc.*, 1910, B. 82, 217) has examined tetrachlorethane and trichlorethylene, and his values (*cf.* Gowing Scopes, ANALYST, 1910, **35**, 238) for some of the properties of these are given below with figures for carbon tetrachloride and carbon disulphide:

		Refractive Index.	Boiling-Point.
CCl_4, carbon tetrachloride ...	$\frac{12{\cdot}3^\circ}{4^\circ}$ 1·6095	(12·3° C.) 1·4656	76–77° C.
$(CHCl_2)_2$, tetrachlorethane ...	$\frac{17^\circ}{17^\circ}$ 1·6013	(17° C.) 1·495587	147·2° C.
CCl_2, [trichlorethylene... ... $CHCl$,	$\frac{17{\cdot}5^\circ}{17{\cdot}5^\circ}$ 1·4702	(17° C.) 1·479141	87·55° C.
CS_2, carbon disulphide... ...	$\frac{0^\circ}{4^\circ}$ 1·2921	(20° C.) 1·6277	46·0° C.

As even the higher fractions of petroleum oil or coal-tar hydrocarbons seldom have a sp. gr. of 0·9, anything above this figure will, unless such substances as nitrobenzene or carbon disulphide are present, indicate the presence of chloro-compounds; and, conversely, anything below a sp. gr. of 1·47 will indicate the admixture of lighter substances with the chloro-compounds.

In any ordinary case if the oil freed from alcohol, ether, and acetone flashes below 73°F., and contains no carbon disulphide, it must contain "petroleum" in the eyes of the law, since chlorine compounds do not flash themselves, but raise the flashing-point of other liquids.

A mixture of carbon tetrachloride and benzene, when tested in the Abel apparatus, shows the inhibitive effect of the chloro-compound on the inflammability of the benzene vapour in a very pronounced degree. Instead of the bright, almost explosive, flash usually associated with the latter, one observes (probably after the slide has been opened several times), a reddish-coloured flame travel comparatively slowly across the surface of the liquid. No doubt the chloro-compound exerts a double effect in retarding flashing by diluting the inflammable vapour, and also (owing to its vapour density) by preventing the access of air.

From a mixture containing possibly both aromatic and aliphatic hydrocarbons and chloro-compounds of the latter, it is easy to remove aromatic hydrocarbons either by *careful* nitration followed by fractional distillation or by sulphonation. It must be borne in mind that if terpenes have been found in the liquid, the presence of aromatic hydrocarbons to the extent of from 5 to 12 per cent. of the terpenes found is probable, owing to the cymene obtained when turpentines are polymerised. This amount should be deducted from any aromatic hydrocarbons which may be found.

The approximate determination of the amount of paraffins and cycloparaffins in admixture with chloro-compounds can be made by a consideration of the physical properties of the mixture and of its probable constituents. We hope at a later date to be able to propose a fairly accurate method of estimation, but at present do no more than make the above suggestion with the warning that the accuracy of all physical methods is dependent on a qualitative knowledge of the constituents of the mixture.

We think we have shown that the examination of mixtures under the Petroleum Acts requires an amount of knowledge and judgment that places it above the powers of the ordinary "tester" to carry out efficiently, and is work which might more properly be entrusted to the Public Analyst or other chemical officer of the Local Authority administering the Acts.

We have examined several samples of liquids sold as paint removers, and have found them to consist principally of acetone and benzene; smaller amounts of other organic liquids are usually present, and we have identified alcohol, chloro-hydrocarbon derivatives and coal-tar naphtha in different samples. Several liquids labelled "non-inflammable solution" were found to consist of a wax or a mixture of waxes dissolved in carbon-tetrachloride and benzene. We have also detected light petroleum in mixtures sold for lacquering purposes. In these cases it was admixed with amyl alcohol. Amyl acetate and ethyl alcohol were also present in one instance. In some cases the dissolved substance is collodion cotton, and we understand that lacquers of this class are extensively used by silversmiths for protecting articles of silver plate from atmospheric influences.

DISCUSSION.

Mr. ARCHBUTT said that in testing the so-called "non-inflammable" mixtures he had always found it desirable not to rely simply on the fact of their flashing or not flashing in the official apparatus, but to put some of the material into a larger vessel and test it with a naked flame, and also to warm some of it in a fair-sized porcelain crucible and then test it. Under these conditions it was sometimes found that mixtures which did not actually flash in the Abel apparatus were nevertheless far from being uninflammable.

Mr. COSTE agreed with Mr. Archbutt; but the limitations as to what one might do when testing under the Petroleum Acts were very strict. They did not even allow of the correction of the flash-point for barometric pressure. The necessity for such a correction was only discovered by Sir Frederick Abel after the Act of 1881 was passed, and the Act had not since been amended to allow of it.

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A METHOD FOR DETERMINING THE AMOUNT OF INSOLUBLE PARTICLES IN RAW RUBBER.

BY CLAYTON BEADLE AND HENRY P. STEVENS, M.A., PH.D., F.I.C.

(Read at the Meeting, December 6, 1911.)

CONSIDERABLE difficulties exist in separating raw rubbers from insoluble extraneous bodies in such a manner that these may be collected, examined and estimated. The most practical manner is to determine the loss on washing, but there is some difficulty in collecting the material removed in the process. For some purposes it is useful to reverse the operation and to remove the rubber and that which properly belongs to the rubber, and to collect, examine, and weigh the extraneous matter. Washing the material on the rolls in the ordinary way is the best procedure from the point of view of the examination of the rubber, but something is wanted, to supplement this test, and which will permit the examination of those impurities which are discarded and thrown away in this process. Moreover, the samples are at times too small to enable this to be done with advantage on ordinary washing rollers; further, with some kinds of scrap and tacky rubbers, the process of removal of the insoluble impurities is not sufficiently complete without overworking the rubber, and there are many cases in which the whole of the mechanical impurities are not removed even after prolonged treatment on the washing rollers. A very small amount of rubber is required for the test about to be described, and the smallness of the amount may be used as an argument against its adoption, because it may be suggested that so small a quantity cannot be taken to represent the bulk. This difficulty can be overcome by proper sampling. When the whole mass is sufficiently homogeneous, a portion of the crepe is taken for analysis. The treatment on the rolls should be as short as possible to avoid disintegration of the particles that exist in the rubber.

The insoluble constituents may be separated by dissolving the rubbers in benzene and allowing the insoluble constituents to settle out from the solution, or by filtration, but the drawback is the viscosity of the solution obtained, which hinders the sedimentation or filtration of the particles. This applies even to very dilute solutions of high-grade rubbers.

Many raw rubbers, particularly those in which the determination of the insoluble solid particles would be of most value, are difficultly soluble in naphtha or other solvents without mastication, which, however, has the disadvantage of disintegrating the insoluble matter. The insoluble matter may be estimated by decanting the top half of the solution as recommended by Schidrowitz, evaporating off the naphtha, and determining the weight of rubber. Knowing the total amount of rubber taken in the first instance and subtracting from this twice the weight of dry rubber obtained from the clear solution, the difference is the weight of the sediment. This latter, however, cannot be separated without further washing and filtration.

The method we have adopted is a modification of that employed in the case of vulcanised rubber goods. To bring about a separation of mineral in such materials, it is customary to heat the finely-ground rubber with an organic liquid of sufficiently high boiling-point to such a temperature as to bring about depolymerisation of the rubber and subsequent solution of the latter in a partly decomposed state. For this purpose nitro-benzene was introduced by Weber and subsequently nitro-naphthalene. Both these substances have disadvantages—the poisonous nature of the vapours of the former, and the fact that the latter is solid at ordinary temperatures. Subsequently xylol was suggested, the rubber and liquid being heated under pressure, and a further advance on this was a suggestion for using high-boiling petroleums which render an autoclave unnecessary. Phenetol has also been used, and we have found it very convenient for the purpose. Now it is well known that fully vulcanised mechanicals are much more difficult to treat by this process than soft rubbers. The latter, as might be expected, yield much more easily to the solvent action of the fluid. Raw rubbers are still more easily acted upon, and a very short treatment at a relatively low temperature is sufficient to dissolve the rubber and so alter its character that, on diluting the product with naphtha, the viscosity of the solution is very much lower than that of the original raw rubber when dissolved in naphtha.

For treating raw rubbers we have used nitro-benzene, high-boiling petroleum, and phenetol (of the three we prefer phenetol). One grm. of the finely-cut rubber is placed in a test-tube with 5 to 10 c.c. of the solvent. The temperature should be raised very slowly, so as to give the rubber plenty of time to swell. It is best to heat for about half an hour at 100° C., then to gradually raise the temperature to about 140° C. for one to one and a half hours. The rubber swells until the whole mass is a jelly, then, on gradually heating to a certain point, a rather sudden change takes place, and the thick, jelly-like mass becomes rapidly thin and liquid. During the heating a peculiar smell is given off, resembling hot fat or roasting meat. On cooling, the whole is poured into about 100 c.c. of benzene and the insoluble matter allowed to settle. We find it advisable to wash once or twice with benzene by decantation before transferring to a filter-paper and washing. In some cases, however, it is preferable to remove the soluble matter by repeated decantation with naphtha, evaporating off the last traces and weighing the residue in a tared dish.

We obtained the undermentioned results. The first column shows the total sediment, the second the amount of ash obtained by incinerating the sediment, and the third column the organic matter by difference. The most useful figure, according to our opinion, is the one given in the first column, but the others are useful in determining how far these insoluble particles are due to organic and how far to mineral constituents. We have also found that a repeat test gives good agreement:

	1. Grms.	2. Grms.	3. Grms.
Hard cure Para unwashed as received ...	1·50	0·66	0·84
,, ,, ,, washed and dried ...	0·36	0·20	0·16
Negro Head...	0·48	0·26	0·22
Plantation crepe	0·29	0·05	0·24
Castilloa	6·80	3·78	3·02
,, creped	5·90	1·10	4·80
Wild rubber A	6·88	2·85	4·03
,, B	6·46	1·06	5·40
Gutta...	0·80	0·40	0·40

The separated material is available for microscopic examination; and as it is structurally uninjured by the treatment, it may be possible, where it is considered of service, to determine the nature of the insoluble material. The unwashed hard-cured rubber yielded flocculent material of a membranous character which agglomerated on drying. The washed hard-cured rubber showed masses of disintegrated material, and in many cases where the sediments were examined under the microscope it would appear that they were derived from the bark of the tree. The samples of hard-cured rubber and Negro Head were obtained washed and dried from one of the leading manufacturers. The two samples of Castilloa came from the same estate. From their appearance it was evident that they were not by any means free from impurities.

The method may be employed as a useful check on the washing of rubber. As an instance, two samples of scrap crepe gave by this method (A) 1·7 and (B) 2·5 per cent. of insoluble residue. On rewashing, the amount of residue was reduced to 0·7 and 1·4 per cent. respectively. An examination of the material removed in A showed a mixture of sand with a little bark, and in B, bark only. The ash in the washed rubbers amounted to 0·7 and 0·35 per cent. respectively. As the insoluble impurities in A consisted mostly of sand and grit, with very little bark, A presents a case in which the ash determination gives a fair idea of the percentage of foreign matter in the rubber. In B the foreign matter consisted almost entirely of bark débris. In such a case the ash determination gives no idea as to the percentage of insoluble particles. A consisted of so-called "tree scrap" washed and creped, produced by the drying down of latex on the tapping surface after the flow of latex has ceased. The rubber is easily peeled off the wood in a thin strip. There is no reason why such rubber should be contaminated with bark chips to any great extent, but it may easily pick up a little sand and grit. Rubber B consisted of "bark scrap"; that is, thin shreds of bark removed by the tapping knife, collected, and macerated wet. The loose pieces of bark were for the most part washed away during the maceration process. Such rubber would naturally tend to retain a little of the bark even after prolonged washing.

It is evident, therefore, that an estimation of ash may be very misleading as regards the amount of insoluble impurities in a sample of rubber. The method we have outlined, although more troublesome than the determination of ash, is nevertheless one which can be carried out fairly rapidly and gives more accurate information as to the character and amount of the insoluble impurities.

DISCUSSION.

Dr. SCHIDROWITZ asked what advantage was possessed by nitrobenzene over benzene as a solvent in this case. He presumed that it was a question of rapidity of solution. He should like to know how the insoluble matter was weighed—whether directly or indirectly—because this was really the most difficult point in the determination.

Dr. RIDEAL said he gathered that with nitrobenzene the whole mass became semi-solid, whereas with benzene it remained fluid. From the figures given it seemed to him that the quantities of insoluble organic and inorganic matters in some of these rubbers were very small, and that after all it was the determination of soluble organic and inorganic matters that might have a value in the future. Since rubber from the same species varied in composition from month to month, according to climatic conditions, etc., to so great an extent as to affect the relative price, it seemed unlikely that these small differences in the insoluble matter were to be regarded as of any great commercial importance.

Dr. SCHIDROWITZ said that in his experience the determination of insoluble matter was one of the most valuable that could be made in rubber—a good deal more valuable, probably, than the determination of nitrogen.

Mr. CLAYTON BEADLE said that the first action of the solvent was to produce a gel, but as the heat was increased the whole mass became perfectly limpid. These insoluble particles were not rubber, but were derived from the bark or from other sources foreign to the rubber itself. In the case of impure rubbers, the insoluble particles were to a large extent removed by the manufacturer during the process of washing, etc. If the rubber was sufficiently transparent, the particles could be seen in it by the naked eye. It was first discovered by Spence that protein in rubber became insoluble when the rubber was treated with benzene and allowed to remain quiescent for a sufficient length of time, and this has since been confirmed by ourselves and other observers. The process described, however, did not leave the proteins in the insoluble residue. The advantage of using such substances as nitrobenzene, nitronaphthalene, or phenetol and other bodies of sufficiently high boiling-points, was that the temperature obtainable enabled one to bring about depolymerisation, so that the liquid was rendered less viscous, from which liquid, after dilution with benzene, the insoluble portion more readily settled out. When this thinning down had taken place, the clear liquid, after dilution with benzene, was poured off, and the insoluble matter, after being washed once or twice with benzene, was dried and weighed.

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NOTE ON THE DETERMINATION OF SMALL QUANTITIES OF METHYL ALCOHOL.*

By C. SIMMONDS, B.Sc.

(*Read at the Meeting, December* 6, 1911.)

SMALL proportions of methyl alcohol, especially when in mixture with ethyl alcohol, have hitherto been somewhat difficult to determine readily and accurately. Fairly good approximate results can be obtained by comparative experiments with the well-known method of Riche and Bardy (*Compt. rend.*, 1875, **80**, 1076), or with Wolff's modification of Trillat's process (*Ann. Inst. Pasteur*, 1902, p. 8), but these methods are lengthy and rather troublesome. The process described by Thorpe and Holmes (*J. Chem. Soc.*, 1904, **85**, 1) gives good results when the quantity of methyl alcohol is not too small. It is not well adapted, however, for use when the proportion of methyl alcohol is less than about 2 per cent. of the ethyl alcohol, since the necessary subtractive correction (*loc. cit.*, pp. 2, 3) may in such cases equal or exceed the quantity it is desired to estimate. For determining very small proportions of methyl alcohol the method is quite inapplicable.

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* Communicated with the sanction of the Government Chemist.

In such cases satisfactory determinations can be made by applying the principle of colorimetric comparison to Denigès' process for the detection of methyl alcohol (*Compt. rend.*, 1910, **150**, 832).

The possibility of thus using the process is indicated by Denigès (*loc. cit.*, p. 833). The object of the present note is to give the procedure which the writer finds most suitable for utilising the reaction quantitatively in general analytical work—as, for example, in examining spirituous beverages, medicinal tinctures, flavouring essences, and so forth.

The alcoholic mixture is first purified, where necessary, either by the method of Thorpe and Holmes (*J. Chem. Soc.*, 1903, **83**, 314), or by other suitable means. It is then diluted with water or mixed with ethyl alcohol, as the case may require, until it contains 10 per cent. of total alcohol by volume.

To 5 c.c. of this prepared liquid contained in a wide test-tube are added 2·5 c.c. of permanganate solution (2·0 grms. $KMnO_4$ per 100 c.c.), and then 0·2 c.c. of strong sulphuric acid. When the reaction has proceeded about three minutes, 0·5 c.c. of oxalic acid solution is added (9·6 grms. crystallised acid per 100 c.c.). On shaking, the liquid becomes clear and nearly colourless. One c.c. of strong sulphuric acid is now run in and well mixed with the solution, which is finally treated with 5 c.c. of Schiff's reagent. A violet colour is developed in the course of a few minutes unless mere traces of methyl alcohol were present, when twenty or thirty minutes may be required.

This colour is due, of course, to the reaction of the fuchsine solution with formaldehyde, produced by the oxidation of the methyl alcohol. A sufficient quantity of sulphuric acid is present to prevent the development of colour with any acetaldehyde formed from the ethyl alcohol during the oxidation.

A preliminary experiment carried out as described serves to detect the presence of methyl alcohol (if this is not already known) and to give some idea of the quantity. According to the indications thus obtained, another part of the prepared liquid is further diluted, if necessary, with ethyl alcohol of 10 per cent. strength until it contains from 0·001 to 0·004 grm. of methyl alcohol in 5 c.c.; and the experiment is repeated side by side with two or more standards for comparison. These contain 0·001, 0·002, 0·003, etc., grm. of methyl alcohol in 5 c.c. of 10 per cent. ethyl alcohol. The colours produced are compared in small Nessler-tubes (25 c.c.) or in a suitable colorimeter.

With properly sensitive Schiff's reagent, 0·0003 grm. methyl alcohol in the 5 c.c. of liquid taken is readily detected. The best depths of colour for comparison, however, are given by the formaldehyde produced in the manner described from quantities of 0·001 to 0·004 grm. of methyl alcohol.

It is convenient to keep a standard solution (1 grm. per litre) of methyl alcohol in 10 per cent. ethyl alcohol. This is diluted as required with 10 per cent. alcohol to form the standards for comparison. This proportion of ethyl alcohol (10 per cent.) is a suitable strength for general work, as the distillates ordinarily obtained are stronger, and can thus be diluted down instead of having to be concentrated.

The process has the advantages of (1) being rapidly executed, (2) requiring only a small quantity of material, and (3) being directly applicable to weak distillates. The degree of accuracy obtainable is shown by the following results of a typical series of experiments:

GRM. METHYL ALCOHOL PER 100 C.C.

Present.	Found.
0·005	0·004
0·028	0·029
0·044	0·046
0·072	0·072
0·100	0·104
0·500	0·492
1·000	0·968

Formaldehyde, of course, must be absent from the unoxidised solution of alcohols, or else its effect must be determined and allowed for. Glycerol must also be absent.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Detection of Caramel in Fermented Beverages. V. Pasquero and A. Cappa. (*Gazz. Chim. Ital.*, 1911, **41**, 349-358.)—A method of detecting caramel in beer and wine is based upon the distillation of the furfural in the caramel. The results obtained with fifteen samples of different kinds of Italian beer and thirty-one samples of red and white wine showed that although traces of furfural were separated by direct distillation of the sample, the distillate obtained when the liquid was neutralised with magnesium carbonate was quite free from furfural. On the other hand, when caramel was added to each of these neutralised samples the distillate invariably contained furfural. The test is applied by neutralising free acids in the beer or wine by the addition of magnesium carbonate, and distilling 100 c.c., after the addition of a little pumice stone, until 75 c.c. have passed over. The distillate is made up to 100 c.c. and 20 c.c. thereof shaken with 2 c.c. of pure glacial acetic acid, and 5 drops of colourless aniline in a colorimetric cylinder. If the sample contained any caramel, a rose coloration will be produced within fifteen minutes. By means of this test the presence of caramel was detected in three commercial samples of beer and in samples of Marsala and Coronata wine. C. A. M.

Clavicepsin: A new Glucoside in Ergot of Rye. F. M. Zuco and V. Pasquero. (*Gazz. Chim. Ital.*, 1911, **41**, 368-374.)—From 1 to 2·5 per cent. of a new glucoside, termed "clavicepsin," was separated from different samples of ergot of rye by extraction with 95 per cent. alcohol. When purified by recrystallisation from water it formed acicular crystals melting at 91° C., which were readily soluble in water. After losing its water of crystallisation at 100° to 105° C., it melted at 198° C. It had $[\alpha]_{D20} = +142{\cdot}27°$, and yielded an osazone melting at 205° C. Its elementary composition corresponded with the formula $C_{18}H_{34}O_{16} + 2H_2O$. It did not reduce Fehling's solution until hydrolysed by acids, when it was decomposed into 2 molecules of dextrose and 1 of mannite. C. A. M.

Method for Separating Mixtures of the Seven Coal-Tar Dyestuffs allowed (U.S.A.) to be employed for colouring Foodstuffs. T. M. Price. (*U.S. Dept. Agric. Bureau of Animal Industry, Circular No.* 180; through *Chem. Zentralbl.*, 1911, II., 1556.)—The dyestuffs which are permitted in the United States to be employed for colouring sausage-skins, etc., are amaranth, ponceau 3 R, erythrosin, orange I, naphthol yellow S, light green SF yellowish and indigo-disulphonic acid. An investigation of their solubility reactions led to the following method of separation: About 0·2 grm. of the mixture is triturated with a supersaturated solution of ammonium sulphate, filtered, and washed with ammonium sulphate solution; the filtrate contains amaranth and a little naphthol yellow S, which may be separated by shaking with ethyl acetate. The residue from the treatment with ammonium sulphate is dissolved in water, acidified with acetic acid, and shaken out with ether, which extracts the erythrosin. The aqueous layer, freed from ether, is salted out hot with sodium chloride; the filtrate contains the light green SF and the naphthol yellow S, the latter being separated from the former by shaking with acetone. The aqueous solution of the dyestuffs precipitated by salt, when shaken three times with ethyl acetate, yields the orange I in that solvent. The aqueous residue is heated to expel the ethyl acetate, cooled, and diluted with a 25 per cent. solution of calcium chloride, a solution of stannous chloride is added, and ponceau 3 R is thus precipitated, whilst the leuco compound of indigo-disulphonic acid remains in solution, and may be re-oxidised by hydrogen peroxide to the blue dyestuff. J. F. B.

Determination of Gliadin or Alcohol-soluble Protein in Wheat Flour. R. Hoagland. (*J. Ind. and Eng. Chem.*, 1911, **3**, 838-842.)—As usually recommended, 70 per cent. alcohol is used for this determination; but the author points out that whether this refers to weight or volume is seldom stated, and does, in fact, considerably influence the results obtained, as has been shown by Shutt (*Bull.* 57, Central Expr. Farm, Ottawa, 1907), who obtains a gliadin number of 47·4 with 70 per cent. by weight alcohol, and 53·4 when using 70 per cent. alcohol by volume. Snyder (*Bull.* 105, Bureau of Chem. U.S. Dept. of Agriculture) quotes Teller as finding the amount of protein extracted by 40-55 per cent. alcohol by volume to be practically constant in amount, and the author defends the use of such strength alcohol as will extract the maximum amount of nitrogen from flour. This strength he finds by experiment to be from 45-55 per cent. by weight; 75 per cent. alcohol by weight extracts about the same amount of protein as distilled water. No advantage is obtained by using hot or boiling alcohol. The method used by the author is as follows: Weigh approximately 2 grms. of flour into an 8-ounce stoppered bottle, add 100 c.c. neutral 50 per cent. (by weight) alcohol, shake in a machine for one hour, centrifuge ten minutes, filter and determine the nitrogen in an aliquot portion of the filtrate by the modified Kjeldahl method. Tables are given of the differences obtainable by varying the method of extraction, and show that as much protein can be obtained by vigorous shaking in a rocking machine for sixty to ninety minutes as when the sample is shaken frequently by hand for three or four hours, and then allowed to stand eighteen hours or less. It is recommended that extraction with 70 per cent. alcohol either by weight or volume be discontinued.

H. F. E. H.

Hydnocarpus Fat. K. Lendrich, E. Koch, and L. Schwarz. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 441-458.)—The fat used in the preparation of a certain class of margarine which produced cases of poisoning (*cf.* ANALYST, 1911, **36**, 65) was found to be identical in its properties with the fats obtained from the *Hydnocarpus* species. The fat is known as cardamom or maratti fat, and consists of fatty acids of the constitution $C_nH_{2n-4}O_2$; from these fatty acids, chaulmoogric acid and hydnocarpic acid have been separated and identified. It does not contain fatty acids of the formulæ: $C_nH_{2n}O_2$ and $C_nH_{2n-2}O_2$; the absence of palmitic acid is noteworthy, as Power and Barrowcliff (*Chem. Soc. Proc.*, 1905, **21**, 175) found this acid to be present in *Taraktogenos* (*Hydnocarpus*) *Kurzii* fat and in *Hydnocarpus anthelmintica* fat, but not in *H. Wightiana* fat. The external appearance of the seeds of *H. Wightiana* and *venenata inebrians* is very similar to that of Ceylon cardamoms, and this is probably the reason why the fat in question is termed cardamom fat. When administered to dogs *Hydnocarpus* fat caused vomiting and convulsions. The poisonous properties of the fat appear to have a certain relation to the presence of an ethylene linkage in the structure of the optically active fatty acids of the fat. The authors do not agree with the statement of Thoms and Muller (*cf.* ANALYST, 1911, **36**, 542) that cardamom (maratti) fat is obtained from *Taraktogenos* (*Hydnocarpus*) *Kurzii*; Thomas and Muller do not state how they separated the palmitic acid found, nor do they appear to have established the identity of the hydnocarpic acid. The authors have also examined the fats obtained from the seeds of *H. anthelmintica* and *H. Alpina*, the fats being extracted by means of chloroform. *H. Alpina* fat consisted of a yellow, butter-like mass, without any particular taste or odour; it gave the following physical and chemical results on analysis: refractometer value, at 40° C., 67·5; acid value, 5·6; saponification value, 209·1; iodine value, 84·5; $[\alpha]_{D20} + 49{\cdot}0$. Its physiological action was similar to that of the other *Hydnocarpus* fats.

W. P. S.

Analysis of Candied Lemon Peel. F. Hartel and A. Kirschner. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 350-353.)—Lemons intended for the manufacture of candied peel are imported packed in brine, the fruit being cut into two parts before being thus pickled. After washing with water, the fruit is boiled for about two

hours, then cooled rapidly in cold water, and the pulp is removed. The peels are next steeped in water for one day, and then air-dried for a day or placed in direct sunlight. They are now immersed in a sugar solution of sp. gr. 1·074, and afterwards in a sugar and starch-syrup solution of sp. gr. 1·115 to 1·383, in which they are allowed to remain for about four weeks; the quantity of starch-syrup employed is from 15 to 25 per cent. of the weight of the total sugars in the solution. The peels are finally glazed with concentrated sugar solution. Twenty samples of candied lemon peel examined yielded the following results: Insoluble substances, 2·89 to 5·22 per cent.; soluble extractives, 60·83 to 79·22 per cent.; acidity (as citric acid), 0·04 to 0·20 per cent.; sucrose, 14·30 to 48·50 per cent.; starch-syrup (anhydrous), 9·45 to 25·30 per cent.; ash, 0·37 to 3·09 per cent.; sodium chloride, 0·02 to 0·09 per cent.
W. P. S.

Detection of Nitric Acid in Watered Fruit Juices. R. Cohn. (*Zeitsch. öffentl. Chem.*, 1911, **17**, 361-363; through *Chem. Zentralbl.*, 1911, II., 1613-1614.)—About 75 c.c. of the juice are made alkaline with sodium hydroxide, evaporated nearly to dryness, and the residue is extracted for several minutes with alcohol at a temperature of 40° C. The residue from the alcohol, which, if it is still syrupy, should again be extracted with alcohol, is dissolved in about 10 c.c. of water and tested for the presence of nitric acid by the diphenylamine reaction. In the case of pure natural raspberry, cherry, and currant juices, a positive reaction was never encountered. In the case of bilberry juice the test cannot be used, since this fruit contains a body which gives a blue coloration with sulphuric acid alone. In presence of bilberry juice the test for nitric acid in the aqueous solution of the alcoholic extract, prepared as above, may be carried out with nitron. In the extracts from pure natural raspberry, cherry, currant, and bilberry juices an acetic acid solution of nitron produces no precipitate; in presence of nitric acid a separation of nitron nitrate soon takes place.
J. F. B.

Changes in the Composition of the Nitrogenous Constituents of Meat-Extracts. A. M. Wright. (*J. Soc. Chem. Ind.*, 1911, **30**, 1197-1198.)—The action of hot water, in presence of the sarco-lactic and other acids of flesh and of salts, on the meat causes a certain amount of hydrolysis, the collagen of muscle-fibre yielding gelatin, and later soluble gelatin and other gelatinoids, and the albumin yielding small quantities of albumoses. During concentration, under a partial vacuum, the amount of organic matter slightly decreases (relatively), while the mineral salts increase, and a small proportion of peptone-like bodies are produced. Concentration in an open pan yields very different results, profound changes taking place. The amount of organic matter is considerably decreased, while there is a corresponding increase of mineral matters. The total nitrogen remains about the same, but the forms in which it occurs are different, about three-fifths of the insoluble and coagulable proteins being converted into soluble nitrogenous compounds. Proteoses and meat-bases are decreased in amount, while peptone-like bodies appear in considerable quantity, and the acidity is notably increased. These peptone-like bodies impart a bitter taste to the extract in the cases of those containing them to the extent of, say, 12 per cent. or over. The "burned" flavour of certain meat-extracts is doubtless due to this method of concentration.

The following analyses illustrate the differences noted. The "open" concentration was carried out by evaporation for forty-eight hours at 212° F., the resulting extract containing 17·6 per cent. moisture. The other method, followed on a portion of the same original liquor, was by a partial vacuum of 15 inches at 180° F. for three and a half hours, the resulting extract containing 18·36 per cent. moisture. All the results have been calculated to a standard basis of 20 per cent. moisture for comparison purposes:

	Extract Liquor.	Concentrated in Open Pan.	Concentrated under Partial Vacuum.
	Per Cent.	Per Cent.	Per Cent.
Moisture	20·00	20·00	20·00
Organic matter	65·60	60·48	63·04
Mineral salts	14·40	19·52	16·96
Acidity as lactic acid	8·00	10·30	8·20
Total nitrogen	7·82	7·92	7·86
Insoluble and coagulable proteins	2·65	1·16	2·63
Proteoses	13·34	12·37	13·08
Peptone-like bodies and polypeptides	nil	8·69	0·31
Total meat-bases	14·19	12·53	13·76
Ammonia ...	0·85	0·80	1·08

A. R. T.

Detection of Saponin and Glycyrrhizin in Lemonades by Means of Vamvakas' and Frehse's Tests. A. Behre. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 498-506.)—The two tests mentioned in the title were found to be unreliable. Vamvakas' test (ANALYST, 1906, **31**, 232), yielded positive results (precipitates with Nessler reagent) with lemonade which was free from saponin and glycyrrhizin, as well as with other samples containing these two substances; the colour of the precipitates was not characteristic and evidently depended on the presence of substances other than saponin and glycyrrhizin. Frehse's test (ANALYST, 1900, **25**, 10) failed owing to the fact that pure saponin is insoluble in ethyl acetate (the solvent recommended for its extraction), and the residue obtained on evaporating the solvent gives no coloration with sulphuric acid. Lemonades containing impure saponin, or so-called saponin, yielded a residue which gave a doubtful reaction with sulphuric acid.
W. P. S.

Estimation of Saponin and Saccharin in Oil Emulsions. E. Carlinfanti and P. Marzocchi. (*Boll. Chim. Farm.*, 1911, **50**, 609-615; through *Chem. Zentralbl.*, 1911, II., 1488-1489.)—One hundred grms. of the oil emulsion are diluted with an equal volume of water, and then gradually treated with 400 c.c. of 95 to 96 per cent. alcohol with constant shaking, which is frequently repeated during two hours, after which it is allowed to stand for twenty-four hours. The clear supernatant liquid is decanted, and the residual fatty mass filtered through linen, again extracted with 60 to 65 per cent. alcohol, and again filtered. The united filtrates are neutralised with sodium carbonate and concentrated to 100 c.c. on the water bath. Aromatic compounds are now extracted with ether, and the residual liquid shaken with 20 grms. of sodium sulphate and 9 c.c. of phenol, this treatment being repeated several times, until the whole of the saponin has been extracted by the phenol. The united phenol extracts are then shaken with a mixture of 100 c.c. of ether, 30 c.c. of water and 5 c.c. of alcohol, and left for twenty-four hours, after which the upper layer containing the saponin is separated and evaporated, and the residue extracted with acetone. The ethereal extract, containing the aromatic compounds, is shaken with water acidified with phosphoric acid, the ether evaporated, and the saccharin estimated in the form of alkali salicylate. C. A. M.

Separation and Identification of Saccharin and Dulcin in Beverages, Foods, and Drugs. S. Camilla and C. Pertusi. (*Giorn. Farm. Chim.*, 1911, **60**, 385-393; through *Chem. Zentralbl.*, 1911, II., 1269-1270.)—The material is treated with an excess of barium hydroxide solution, warmed, filtered, and the filtrate extracted with ether after being acidified with sulphuric acid; the saccharin and dulcin crystallise out when the ethereal solution is evaporated. For the identification of these two sweetening materials in the presence of salicylic acid, the sample is mixed with magnesium oxide, evaporated to dryness, and the residue extracted with acetone containing 10 per cent. of water. The acetone solution is filtered, evaporated, the residue dissolved in water, and the solution extracted with ether; dulcin goes into solution. The aqueous solution is then acidified with sulphuric acid, and the saccharin is extracted by means of a mixture of ether and petroleum spirit Should salicylic acid be present, the aqueous solution containing the saccharin must be evaporated to dryness, the residue dissolved in 10 per cent. sulphuric acid, and the solution treated with an excess of potassium permanganate; after filtration the saccharin is extracted from the filtrate with a mixture of ether and petroleum spirit (*cf.* ANALYST, 1910, **35**, 436).

Alteration of the Acid-Content of Distilled Vinegar when Stored in Wooden Casks. A. Behre. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 506-509.) —The acidity of a cask of vinegar increased in the course of nine months from 7·10 to 9·12 per cent.; the total solids also increased from 0·30 to 0·39 per cent., and the concentration of these constituents was evidently due to the more rapid diffusion of the water through the pores of the wood. Further experiments were made in which acetic acid solutions of various strengths were exposed to the atmosphere in glass vessels. In glass cylinders solutions containing from 7·44 to 61·2 per cent. of acetic acid scarcely altered in strength during ten weeks, but when exposed in flasks having narrow necks the more concentrated solutions remained unaltered, whilst those containing less than 19 per cent. of acetic acid increased in strength to a slight extent.

Considerable changes took place when the solutions were exposed in open beakers; the stronger solutions lost large quantities of acetic acid, whilst the weaker solutions became stronger. Under these conditions, a vinegar containing 3·17 per cent. of acetic acid was found to have 4·68 per cent. after the lapse of four weeks; a solution containing 19 per cent. of acetic acid did not alter in strength; whilst a solution containing 61·2 per cent. lost 2·2 per cent. of acetic acid during this period.

W. P. S.

Estimation of Phosphorus in Wine. J. Dormane. (*Bull. Assoc. Chim. Sucr. Dist.*, 1911, **29**, 63-67; through *Chem. Zentralbl.*, 1911, II., 1376-1377.)—Three samples of Burgundy wine yielded the following results: Total phosphorus (as P_2O_5), 0·231 to 0·461; phosphoric anhydride (P_2O_5), 0·160 to 0·390; phosphoric anhydride *plus* phosphorus in organic combination (as P_2O_5), 0·205 to 0·422 grm. per litre. The total phosphorus was estimated by igniting the total solids of the wine in the presence of calcium oxide and potassium carbonate; the phosphoric anhydride (phosphate) by extracting a further quantity of the total solids with cold, dilute hydrochloric acid; whilst the phosphoric anhydride *plus* the organically combined phosphorus was obtained by boiling the total solids for two hours with concentrated hydrochloric acid. The solutions were then treated in the usual way for the precipitation of the phosphoric acid. It is evident that wine contains phosphorus compounds which are not decomposed by hydrochloric acid.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Detection of Albumoses in Urine. E. H. Fittipaldi. (*Deutsch. Med. Woch.*, 1911, **37**, 1890-1891; through *Chem. Zentralbl.*, 1911, II., 1489-1490.)—From 10 to 20 c.c. of the urine are treated with six times the quantity of absolute alcohol, and the resulting precipitate separated by decantation after twenty-four hours, and dissolved in the smallest possible quantity of 31 to 32 per cent. sodium hydroxide solution. This alkaline solution is tested with a freshly-prepared ammoniacal solution of nickel (equal parts of strong ammonia and 5 per cent. nickel sulphate solution), and in the presence of albumoses or peptones gives an immediate orange-red coloration. The reaction is not obtained with albumins, uric acid, cyanuric acid, xanthin, hypoxanthin, sarcosin, or urobilin. The method may also be used for the detection of albumoses in blood, after the latter has been heated with animal charcoal, cooled, and filtered. The reaction is not given by C-deutero-albumose or A-peptone, but neither do these bodies give the biuret reaction. Albumoses may thus be detected in urine in cases of albuminuria, especially when large amounts of albumin are excreted. This is attributed to the presence of proteolytic enzymes in the urine.

C. A. M.

Application of the Biochemical Method to the Analysis of Uva-Ursi Leaves. A. Fichtenholz. (*J. Pharm. Chim.*, 1911, **4**, 441-446.)—The leaves of *Arctostaphylos Uva-ursi* are the source from which the glucoside arbutin is extracted commercially. The biochemical method, depending on the hydrolysis of the arbutin by emulsin after the extract has first been subjected to the action of invertase, may be applied advantageously to the analysis of the dried leaves. The leaves are first extracted with boiling 90 per cent. alcohol, the extract is evaporated to dryness under reduced pressure, the residue is taken up with water, the solution is clarified with basic lead acetate and filtered, the precipitate being washed on the filter. The excess of lead in the liquid is removed by sulphuretted hydrogen, and the solution is then concentrated at a temperature not exceeding 45° C. under reduced pressure. Alcohol is added, and evaporation continued to dryness. The residue is dissolved in thymolated water, the solution is filtered and polarised; the cupric reducing substances calculated as dextrose are also estimated. If the presence of free hydroquinone is to be feared, this solution should be exhausted by ether; it is then ready for treatment with enzymes. It is first treated with 0·15 grm. of invertase (top-fermentation yeast, treated with 95 per cent. alcohol and dried), and is digested at 15° to 18° C. for about five days. The flask is then placed in a boiling water-bath for twenty minutes, cooled, and the emulsin (0·15 grm.) is added. During the whole period of digestion samples are taken at intervals and polarised, the cupric-reducing value being also estimated. After the action of the invertase, the lævo-rotation of the liquid will have increased, but when the emulsin begins to act the lævo-rotation decreases; the results are calculated on the assumption that under the action of emulsin a decrease of 60′ corresponds to the hydrolysis of about 0·5 grm. of arbutin per 100 c.c. The action may continue over ninety-five days. From the comparative rise in cupric-reducing value, some idea may be obtained of the relative proportion of methyl arbutin also present. J. F. B.

Composition of Hens' Eggs. K. Kojo. (*Zeitsch. physiol. Chem.*, 1911, **75**, 1-2.)—Fluid white of egg was found to contain an average of 87·71 per cent. of water, 11·88 per cent. of organic substances, and 0·4 per cent. of ash; the nitrogen-content was 1·75 per cent., whilst the quantity of dextrose amounted to 0·55 per cent., or 4·64 per cent. calculated on the organic substance. The fluid yolk contained 49·73 per cent. of water, 48·83 per cent. of organic substances, and 1·44 per cent. of ash; 2·49 per cent. of nitrogen, and 0·27 per cent. of dextrose were present. Calculated on the organic substance, the percentage of dextrose was 0·55, a much smaller proportion than was present in the white of egg. Only traces of creatinine were found. W. P. S.

Tests for Ascertaining the Freshness of Milk. W. Morres. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 459-464.)—The alcohol test for milk, which depends upon the fact that milk containing upwards of 8 degrees of acidity (Soxhlet-Henkel) usually curdles when mixed with twice its volume of 70 per cent. alcohol is not always a reliable test of the acidity of milk, as the curdling may be caused by means other than the formation of lactic acid. For instance, milk may be on the point of curdling owing to the presence of rennet produced by bacteria, although it may contain but little free acid. Various strengths of alcohol have been proposed for use in the test, and the author considers that the use of 50 per cent. alcohol will afford useful information. A more stringent test with 68 per cent.

alcohol may be applied when the milk is intended for feeding infants, etc. The coloration produced when milk is treated with a solution of alizarin in 68 per cent. alcohol gives an indication of the kind of fermentation which has taken place in the milk. Should the milk curdle, but the coloration remain lilac or red, the milk contains only rennet products; acid formation is shown by a yellow coloration.

W. P. S.

Relations between the Reductase Test, Acidity, and Alcohol Test in Milk. W. Morres. (*Milchw. Zentralbl.*, 7, 441-445; through *Chem. Zentralbl.*, 1911, II., 1614.)—The alcohol test in combination with alizarin, which the author calls the alizarol test, gives easily and rapidly the most reliable indication of the degree and nature of the decomposition of the milk. A comparison of the reductase test with the alizarol test showed: (1) That the alizarol test is equivalent to the reductase test both as regards the degree of curdling and the change of colour in the case of pure lactic acid fermentation; (2) that the smallest degree of decomposition which the alizarol test will show definitely (8 degrees of acidity) corresponds to a decolorisation time of sixteen minutes at 40° C., corresponding to more than 20,000,000 germs per 1 c.c. In the testing of milk from a hygienic point of view it is desirable to express the results in terms of its contamination with germs. Milk "rich in germs" would be one which decolorises the test in from five to fifteen minutes at 40° C., whilst "very rich in germs" would describe a sample which decolorises in less than five minutes at 40° C. Such milk can be very surely detected by the alizarol test more rapidly than by the reductase test. In the case of samples poor in germs, which require more than fifteen minutes for decolorising the methylene blue, the reductase test is the only practical method for the estimation of the approximate contamination with germs. As to the nature of the bacteria present, the fermentation test, or the fermentation-reductase test, of Orla-Jensen affords further information. Since, in practice, the carrying out of the reductase test at 40° C. presents certain difficulties, the author recommends that the test be performed at about 20° C., and the results calculated at 40° C. by multiplying them by $\frac{1}{4}$.

J. F. B.

New Method for Determining the Tryptic Value of Pancreatin. C. F. Ramsay. (*J. Ind. and Eng. Chem.*, 1911, 3, 822-823.)—The United States Pharmacopœia method for determining the tryptic value of pancreatin is unsatisfactory owing to the uncertainty of the end reaction, and consists in finding the amount of enzyme preparation capable of peptonising milk to such a degree that no coagulation follows the addition of nitric acid. The results depend on the amount of acid used, since a little acid gives no precipitate; more acid will always give a precipitate, and still more acid redissolves it. However long the digestion, acid will always cause a precipitation in peptonised milk. The author's method consists in finding the amount of the enzyme preparation which in fifteen minutes will so peptonise milk that no coagulation or precipitate is formed on the addition of slightly acidified rennin solution. The milk is rendered slightly alkaline with sodium bicarbonate to prevent the pancreatin curdling the milk. The materials required are:

(1) 0·5 grm. pancreatin diluted to a volume of 50 c.c. with distilled water, (2) 900 c.c. milk containing 1·8 grm. of sodium bicarbonate; and (3) 2 grms. of rennin and 1 c.c. of 6 per cent. acetic acid (U.S.P.) added to 50 c.c. of distilled water. The pancreatin is first tested at wide ranges of strength, such as 1 grm. to 700 c.c. milk or 1 grm. to 1,000, and a second series is then carried out over a narrower range. In each of several tubes of 100 c.c. capacity are placed 50 c.c. of the milk; the tubes are placed in a bath at 40° C., and varying volumes of the enzyme preparation are added corresponding to (say) 1 in 600, 1 in 650, 1 in 700, etc. After fifteen minutes' digestion, 5 c.c. of the digested milk are withdrawn, 3 c.c. of the rennin solution added, and the whole well shaken. No precipitation indicates that the casein is all peptonised, and the enzyme is stronger than the dilution tested. The amount of acid in 3 c.c. of the rennin solution will exactly neutralise the amount of bicarbonate in 5 c.c. of the milk; the rennin then acts under favourable conditions, since no precipitate is formed in an alkaline solution. Pancreatin in neutral solutions deteriorates rapidly, and consequently should only be made up immediately before use. The author claims an accuracy of 2 to 3 per cent. for the method, and suggests a standard of 1 to 800 or 1 to 1,000 as being a suitable strength for pancreatin preparations. Commercial samples showed variations of from 1 to 120 to 1 to 1,750.

H. F. E. H.

Estimation of Urea. Desgrez and Feuillié. (*Comptes rend.*, 1911, **153**, 1007-1010.)—The following modification of Bouchard's method, which is based upon the decomposition of urea by Millon's reagent—

$$CH_4N_2O + HNO_3 + HNO_2 = N_2 + CO_2 + NH_4NO_3 + H_2O$$

—is rapid, and requires only simple apparatus. The ureometer is immersed in a cylinder containing chloroform or carbon tetrachloride, and having round its exterior a metal band with a projection, which may be heated. After the lower part of the ureometer has been filled with chloroform, there are introduced successively 1 c.c. of the urine, about 5 c.c. of water, and 8 to 10 c.c. of Millon's reagent, and the chloroform is then brought to 30° to 35° C. by heating the metal projection. Decomposition of the urea is effected within twenty to twenty-five minutes by shaking the ureometer from time to time. As soon as the volume of gas ceases to increase, the tube is transferred to a cylinder of water, and the gas washed with sodium hydroxide and water, after which the tube is removed to a second cylinder of water, where the last traces of carbon dioxide are removed, and a final washing is given before measuring the quantity of nitrogen. If the temperature of reaction is not allowed to exceed 35° C., no decomposition of ammonium salts, uric acid, amino acids, creatin, creatinin, hypoxanthin, xanthin, tyrosin, leucin, or guanin takes place. Allantoin, however, begins to decompose at that temperature; but since human urine contains only about 0·014 grm. of that substance after twenty-four hours, this does not interfere with the applicability of the method to human urine, whatever the case may be with herbivora. The non-dialysable substances extracted from several litres of urine did not yield any trace of nitrogen when treated with Millon's reagent under the conditions of the estimation.

C. A. M.

Urobilinogen of Urine and Ehrlich's Reaction for Aldehydes. H. Fischer and F. Meyer-Betz. (*Zeitsch. physiol. Chem.*, 1911, **75**, 232-261.)—The crystalline urobilinogen which may be separated from pathological urine is identical with hemibilirubin. Non-stabile pyrroles (dimethylpyrrole, hæmopyrrole, phonopyrrole, carboxylic acid, etc.), to which class of substances belong the crystalline blood-pigment derivatives and the bile-pigment derivatives, are converted in the body into urobilin, and all these pyrroles give a reaction with Ehrlich's reagent (*p*-dimethylaminobenzaldehyde in hydrochloric acid solution). After the administration of these substances, a positive reaction does not necessarily denote the presence of urobilinogen and urobilin in urine; at the same time, urine may contain considerable quantities of urobilinogen and fail to give a reaction with the test. The substance which yields the reaction with Ehrlich's reagent is a dipyrrylmethane colouring-matter derived from the leuco-bases. W. P. S.

ORGANIC ANALYSIS.

Champaca Oils. B. T. Brooks. (*J. Amer. Chem. Soc.*, 1911, **33**, 1763-1772.)—A quantity of the yellow flowers of *Michelia champaca* L. yielded 0·37 per cent. of an oil containing phenyl-ethyl alcohol (free and as ester), cineol, iso-eugenol, benzyl alcohol, benzaldehyde, and benzoic and acetic acids, with a small quantity of a fluorescent substance (? methyl anthranilate). The oil had the following characters:

	I.	II.
Sp. gr., 30°/30° C.	0·9040	0·9107
Refractive index $[n]_D$, 30° C. ...	1·4640	1·4688
Ester number	124·0	146·0
Ester number after acetylation	—	199·0

On standing, the oil deposits comparatively large quantities of a crystalline unstable ketone, melting at 165° to 166° C., and optically active ($[\alpha]_D$, 30° C. = $-82·5°$ in alcohol).

The white flowers of champaca (*Michelia longifolia* L.) yielded 0·08 per cent. of an oil free from fluorescence, and containing linalol, the methyl or ethyl ester of methylethyl-acetic acid, and methyl-eugenol. No crystalline ketone was present. The oil had the following characters:

Sp. gr., 30°/30° C.	0·897
Ester number	180
Refractive index $[n]_D$, 30° C.	1·4470

The flowers of both species of *Michelia* contain an oxidase. A. R. T.

Behaviour of Invert Sugar in the Presence of Alkali and Hydrogen Peroxide. A. Jolles. (*Biochem. Zeitsch.*, 1911, **36**, 389-393.)—When a 2 per cent. invert sugar solution in $\frac{4}{5}$ N sodium hydroxide solution is treated with an excess of hydrogen peroxide, and heated to a temperature of 37° C., the optical rotation of the sugar diminishes gradually. After an hour or so the rotation becomes *nil*, then slightly dextro-rotatory, and finally *nil* again. This is due to the lævulose being more rapidly oxidised than the dextrose. W. P. S.

Analysis of Lactic Acid. A. A. Besson. (*Chem. Zeit.*, 1911, 35, 1209-1210.) —In the estimation of anhydride in commercial lactic acid it is sufficient to add an excess of 2·5 c.c. of $\frac{N}{1}$ sodium hydroxide solution to the neutralised lactic acid (1 grm.), allow the mixture to stand for ten minutes, neutralise the alkali, and to heat the solution just to boiling with an excess of 1 c.c. of $\frac{N}{1}$ sulphuric acid in order to obtain the quantity of anhydride present (*cf.* ANALYST, 1911, **36**, 235, 556).

W. P. S.

Determination of Malic Acid. P. B. Dunbar and R. F. Bacon. (*J. Ind. and Eng. Chem.*, 1911, 3, 826-831.)—The authors have worked out a polarimetric method for the estimation of malic acid on similar lines to that described by Yoder (ANALYST, 1911, **36**, 498), based on the increase of rotation produced by the addition of uranyl acetate. The acidity of the solution is first titrated on a separate portion with standard alkali in presence of phenolphthaleïn; 75 c.c. of the solution are then placed in a 100 c.c. flask, together with the requisite quantity of standard alkali; if dark in colour, 10 c.c. of alumina cream are added, the liquid is made up to the mark and filtered. Twenty-five c.c. of the filtrate are treated with sufficient powdered uranyl acetate, so that a small amount remains undissolved after two hours; 2·5 grms. are generally sufficient, unless the quantity of malic acid is large. After two hours with frequent shaking, the solution is filtered and polarised, if possible, in the 200 mm. tube. The result is called reading (1). The remainder of the original filtrate is treated with powdered lead acetate until no further precipitation results. The liquid is cooled in ice, and filtered clear; the filtrate is rewarmed to the ordinary temperature, and a crystal of lead acetate is added to determine whether precipitation is complete; if so, the excess of lead is removed by anhydrous sodium sulphate, and the liquid is filtered and polarised, the result being called reading (2). The lead acetate precipitates the malic acid, but as lead malate is soluble in excess of lead acetate, care is necessary in performing this operation. Solutions which contain less than 10 per cent. of reducing sugar may be polarised without treatment with lead. If reading (2) is negative, a portion of this solution is treated with uranyl acetate as above described and polarised; this is called reading (3). If (2) is positive, reading (3) need not be made. All readings are made with white light on the Ventzke scale, and calculated for the 200 mm. tube. If reading (3) is less numerically than reading (2), reading (3) is accepted, otherwise reading (2) is used. Then the algebraic difference between reading (2) or (3), whichever be used, and reading (1) is multiplied by the factor 0·036, and the product represents the percentage of malic acid in the solution polarised. For this determination the most favourable limits of concentration lie between 0·2 and 2·5 per cent. of malic acid; the error seldom amounts to more than 5 per cent. of the malic acid present. Reading (3) is intended to correct the slight lowering effect of uranyl acetate on the rotation of the sugars. If less than 10 per cent. of reducing sugar and more than 0·25 per cent. of malic acid be present, reading (3) may be dispensed with; in presence of large amounts of sucrose, reading (2) gives better results than reading (3). The method cannot be used for the estimation of malic acid if *d*-tartaric acid be also present.

J. F. B.

Fluorescence Test for Mineral and Rosin Oils. P. H. Walker and E. W. Boughton. (*J. Ind. and Eng. Chem.*, 1911, **3**, 816.)—The Outerbridge method for the detection of mineral and rosin oils in fatty oils is based on the observation that the fluorescence of mineral and rosin oils is greatly magnified by performing the examination by the light of an enclosed electric arc, and that samples which appear non-fluorescent in sunlight become strongly fluorescent when observed in the proper light. The test may be made quantitative by comparison with a set of standards. The authors have confirmed the fact that the light of an enclosed arc has the effect described, but they show that fluorescence in an oil does not necessarily prove the presence of mineral or rosin oil. Out of sixteen samples of linseed oil of authenticated purity, only eight showed no fluorescence, three showed slight fluorescence, and five showed marked fluorescence. A number of other samples of fatty oils of known purity, some having been cold-pressed from the seed, showed marked fluorescence, in some cases as strong as that of many mineral oils. A pure linseed oil which showed no fluorescence was heated to 300° C., and, after cooling, was found to be strongly fluorescent. The same occurred with a pure olive oil after heating. In the case of turpentine oils also, many show no fluorescence; some known to contain mineral oil likewise show no fluorescence, whilst others, certainly pure, show marked fluorescence.

J. F. B.

Catalytic Action of Light on the Oxidation of Phenolphthalin to Phenolphthaleïn. G. Rossi. (*Giorn. Farm. Chim.*, **60**, 433-436; through *Chem. Zentralbl.*, 1911, II., 1659.)—Phenolphthaleïn is reduced by nascent hydrogen to phenolphthalin. The reversed reaction is used by the author for the detection of blood. Hydrogen peroxide is employed as the oxidising agent; any blood which may be present acts as a catalyser; in the absence of blood the oxidation is extremely slow and only takes place after some days. The procedure is as follows: An alkaline solution of phenolphthaleïn is decolorised by shaking with zinc dust, the solution is filtered and added, together with a few c.c. of 12 per cent. hydrogen peroxide, to the liquid to be tested. If blood be present, a pink or red coloration will be developed within three hours. The prolonged action of light will also bring about a similar acceleration of the oxidation of the phenolphthalin. According to the author's experiments the light of an electric arc will cause the liquid in a sealed tube to turn red within two hours.

J. F. B.

Colorimetric Estimation of Phenols in Effluents. H. Bach. (*Zeitsch. anal. Chem.*, 1911, **50**, 736-740.)—Small quantities of phenols in effluents—*e.g.*, from coke by-product plants, etc., may be estimated colorimetrically, in absence of salicylic acid, by means of Millon's reagent. As a preliminary test, 10 c.c. of the effluent, 0·2 c.c. of Millon's reagent, and 0·1 c.c. of strong nitric acid are heated to boiling-point in a test-tube, in such a way that none of the liquid spurts up. If a faint pink or pale red coloration is developed, the effluent is fit for the test; if it shows deep red it must be suitably diluted, and if only a faint yellowish-colour is developed, 100 c.c. of the water are treated with 5 c.c. of concentrated sulphuric acid, and 20 c.c. are distilled off, of which 10 c.c. are tested. If the last case indicates that a concentration of the phenol is required, 1 litre of the effluent is made alkaline with 5 to 10 c.c. of strong caustic potash lye and evaporated to 50 c.c. The residue is placed in a flask, cooled, acidified with 10 to 20 c.c. of strong sulphuric acid, diluted to 150 c.c., and 100 c.c are distilled over; the phenol is thus concentrated ten times. For the colorimetric test, the above-mentioned quantities of the effluent and reagents are heated in a test-tube as described; if the coloration is yellowish to pale pink, 0·2 to 0·7 mgrm. of phenol is present in 10 c.c.; if pale pink to medium red, 0·8 to 1·8 mgrm.; and if medium to deep red, 1·8 to 3 mgrms. These colours change on cooling, but the above indications are sufficient for the preparation of the standard tests, which are made up in a similar manner with a solution containing 0·1 mgrm. of pure phenol per 1 c.c. No comparisons are made until all the tubes are quite cold (*i.e.*, after half an hour), when the colorations become stable and remain fixed for several hours. Comparisons should be made against a white ground both by reflected and transmitted light. With some practice, the quantity of phenol may be estimated with an accuracy of a few mgrms. per litre, unless the effluent is extremely dilute. J. F. B.

Estimation of Water in Soap. R. M. Fitzpatrick. (*Chem. News*, 1911, **104**, 247.)—One grm. of the soap is treated with 50 c.c. of absolute alcohol, a fragment of porous tile added, and the liquid heated on a water-bath under a reflux-condenser till solution is complete. After partial cooling, the liquid is filtered through filter-paper, the soap, etc., remaining in the flask being transferred to the filter by the addition of 20 c.c. of absolute alcohol and warming, and the residue on the paper also washed with absolute alcohol. The residue on the paper is dried and weighed, and the amount of alcohol-insoluble matter thus found. Five grms. of anhydrous sodium sulphate are next added to the cold filtrate, and the closed flask allowed to stand for at least twelve hours. If the solution shows any tendency to form soap jelly, more alcohol must be added before the sodium sulphate is put in. The solution, after standing, is filtered into a flask weighed with a small portion of porous tile, and the sodium sulphate washed several times with warm absolute alcohol. The alcohol is evaporated off, the residual soap dried for fifteen minutes at 100° C. and weighed. The weight of anhydrous soap, *plus* the weight of alcohol-insoluble matters, subtracted from the weight of soap taken, gives the weight of water in the soap. A. R. T.

Qualitative Reactions of Vegetable Tannins. E. Stiasny and C. D. Wilkinson. (*Collegium*, 1911, 318-324, 325-333; through *Chem. Zentralbl.*, 1911, II., 1384-1386.)—The behaviour of some thirty-four different tannin solutions towards various reagents is described; amongst the latter were ammonium acetate, tartar emetic, lead acetate, bromine, copper sulphate and ammonia, and formaldehyde.

INORGANIC ANALYSIS.

Simplification of Gravimetric Analysis. E. Murmann. (*Zeitsch. anal. Chem.*, 1911, **50**, 742-747.)—The author describes means of simplifying the operations of filtering, washing, and drying precipitates. Slimy precipitates may be mixed with fine rice starch or fine paper pulp before filtering. J. F. B.

Detection of Fluorides. E. Rupp. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 496-497.)—The ash of the substance to be tested for the presence of fluorides is moistened in a platinum or lead crucible with three drops of water and 1 c.c. of sulphuric acid is added. The crucible is loosely closed with a rubber stopper through which passes a piece of glass rod; the lower end of the latter is moistened with a drop of water, and the crucible is heated on a water-bath for about twenty minutes. Should fluorides be present, the rod becomes coated with a film consisting of silica, sodium silicofluoride and calcium silicofluoride, the drop of water having evaporated during the heating. When only traces of fluoride are suspected to be present, the portion of the glass rod below the stopper may be covered with a piece of rubber tubing, leaving only the end of the rod exposed. In this way the action of the hydrofluoric acid is concentrated on a small surface of the glass.

W. P. S.

Estimation of the Ferrous Ion by Means of Iodine. G. Romijn. (*Chem. Zeit.*, 1911, **35**, 1300.)—Ferrous salts may be oxidised to ferric by means of iodine, provided the reverse reaction be inhibited by suppressing the ferric ion in the solution as it is formed. This may be done by the addition of sodium acetate or tartrate, but the reaction is a slow one, and the complex ferric salts produced are highly coloured. According to the author's observations, sodium pyrophosphate serves a similar purpose; the reaction is complete in five minutes, and the ferric sodium pyrophosphate complex is nearly colourless; sufficient acid must be present to insure that the greater portion of the salt is in the state of the acid salt $Na_2H_2P_2O_7$, whilst a little still remains as the unchanged neutral salt. The iodometric method is applicable to cases where ferric salts and organic matters exist in presence of the ferrous salt to be estimated, also for the analysis of drugs, such as Blaud's pills and ferric iodide syrups, where permanganate cannot be used. The ferrous compound to be analysed is dissolved in such a quantity of sulphuric or hydrochloric acid that the quantity of free acid remaining is equivalent to about 20 c.c. of $\frac{N}{1}$ acid. To this solution are added 20 c.c. of $\frac{N}{10}$ iodine solution and 5 grms. of powdered sodium pyrophosphate; the liquid is rotated until the salt is completely or nearly dissolved. After five minutes the excess of iodine is titrated back with $\frac{N}{10}$ thiosulphate. The results are somewhat lower than those obtained with permanganate (97·5-99 per cent.), probably owing to the oxidation of some of the ferrous pyro-phosphate by the air. J. F. B.

Estimation of Small Quantities of Iron in Organic Substances. F. Jahn. (*Zeitsch. physiol. Chem.*, 1911, **75**, 308-338.)—The following process is recommended for the estimation of iron in cases where only a few mgrms. of the latter are present: The organic substance is heated with sulphuric and nitric acids until all carbonaceous matter has been destroyed and the excess of nitric acid has been expelled. The solution is then diluted with water, boiled, 5 c.c. of concentrated sulphuric acid are added, and the warm solution is rinsed into a flask with water free from dissolved oxygen. Carbon dioxide is now passed into the flask while the contents of the latter are cooled to a temperature below 10° C.; 5 c.c. of 40 per cent. potassium thiocyanate solution are added, and the solution is then titrated with titanous chloride solution. A very small excess of the latter is added, and this excess is titrated back with a standard iron solution. The titanous chloride solution must be kept in a bottle under an atmosphere of carbon dioxide and standardised against the iron solution before and after each estimation. The standard iron solution is best prepared from pure ferric oxide (Kahlbaum). Particular care should be taken that the acid used in the process be free from iron; some 500 c.c. of the nitric or hydrochloric acid should be evaporated to a volume of 10 c.c., then heated with pure sulphuric acid until the latter fumes, and the residual solution, after dilution, tested with thiocyanate. Nitric acid free from iron frequently yields a red coloration with thiocyanate, when tested directly, owing to the presence of traces of nitrous acid. W. P. S.

Gravimetric and Volumetric Methods for the Analysis of Mercury-ammonium Chloride. G. A. Sutterheim. (*Pharm. Weekblad,* 1911, **48**, 1085-1087; through *Chem. Zentralbl.*, 1911, II., 1378-1379.)—A quantity of about 0·1 grm. of the substance is boiled with a small volume of water, 0·5 grm. of potassium cyanide, and 5 c.c. of sodium hydroxide solution until all the ammonia has been expelled. Ten c.c. of formaldehyde solution are then added, the mixture is heated for some time, and the separated metallic mercury is collected, washed successively with water, alcohol, and ether, and weighed. In the volumetric process, 0·2 grm. of the substance is heated with 5 c.c. of water and 3 c.c. of sodium hydroxide solution until the ammonia has been removed, the mixture is cooled, an excess of $\frac{N}{2}$ potassium cyanide solution is added, and, when the mercuric oxide has dissolved completely the solution is nearly neutralised with nitric acid, using phenolphthaleïn as indicator. The uncombined cyanide is then titrated with $\frac{N}{10}$ silver nitrate solution.

New Reaction of Nitrous Acid. W. Vaubel. (*Chem. Zeit.*, 1911, **35**, 1238.)—A sensitive test for nitrites is based upon the fact that mineral acid salts of amino compounds react with nitrites to form aminoazobenzene, which, according to the quantity, either separates as a brown precipitate or causes a yellow turbidity or coloration. Thus, on adding aniline hydrochloride to water containing nitrites, the reaction is produced, and in this way it is possible to detect as little as 0·00035 per cent. of nitrite by the yellow coloration, which, however, in the case of so dilute a solution, takes several hours to develop. C. A. M.

Estimation of Perchloric Acid in Certain Perchlorates. H. Golblum. (*Zeitsch. anal. Chem.*, 1911, **50**, 741-742.)—The method for the estimation of perchlorates, which depends on their conversion into chlorides, when strongly heated with ammonium chloride, is only applicable to the perchlorates of alkali metals; in other cases it fails owing to the decomposition of the resulting chlorides when heated. The author has devised an electrolytic method, which he has employed successfully in the analysis of nickel and cobalt perchlorates, in order to determine the relations between acid and base in these salts. It is based on the fact that the ion of perchloric acid is the last stage in the anodic oxidation of chlorides, and is therefore stable towards complete electrolysis when the metals are deposited on the cathode. Simple titration of the residual liquid with phenolphthaleïn as indicator should therefore afford a quantitative estimation of the perchloric acid, provided other chlorine compounds, derived from any possible decomposition of the perchloric acid, be absent. The results obtained for the metals were very satisfactory. In order to obtain adherent deposits, a platinum wire-net cathode was employed, and a weak current of only 0·1 ampère at the ordinary temperature; the terminal voltage was 10 to 12 on account of the high resistance. In order to decompose 0·2 grm. of the salt dissolved in 300 c.c. of water, it was found necessary to pass the current for seventy hours. J. F. B.

Volumetric Estimation of Small Quantities of Potassium. L. T. Bowser. (*J. Amer. Chem. Soc.*, 1911, **33**, 1752-1757.)—The author has adapted the qualitative cobaltinitrite method previously described by him (ANALYST, 1911, **36**, 606) to the determination of small quantities of potassium, the precipitate of dipotassium sodium cobaltinitrite being filtered and titrated in acid solution with dilute permanganate.

The potassium solution is evaporated nearly to dryness, cooled, and 2·5 c.c. of the sodium cobaltinitrite reagent and 10 c.c. of acid-alcohol (equal parts of glacial acetic acid and 95 per cent. alcohol) added, the mixture being well stirred. The precipitate is filtered through asbestos under suction, only very short contact with the reagent (half an hour) being allowed. The asbestos filter is laid on a perforated aluminium plate in a tube 2 inches long by ½ inch wide. The excess of reagent is removed from the precipitate by washing with acetic acid (20 per cent.). The asbestos and precipitate are transferred to a beaker, using a few c.c. only of water, an excess of 0·005 N potassium permanganate added, the liquid boiled, acidified with exactly 1 c.c. of diluted sulphuric acid (1 : 1), and the excess of permanganate almost immediately titrated at boiling temperature by means of 0·005 N oxalic acid. A blank experiment, to allow for the permanganate used up by the asbestos and sulphuric acid, must be made in each case, and the same asbestos then made into a filter and used for the determination. One c.c. of the permanganate solution is equivalent to 0·000043 grm. K. The presence of sodium, calcium, and magnesium in moderate amounts is without effect upon the results, which are fairly accurate. A. R. T.

Estimation of Sulphur in Pyrites. A. Heczko. (*Zeitsch. anal. Chem.*, 1911, **50**, 748-753.)—For the estimation of the available sulphur in pyrites, Dennstedt's combustion method (ANALYST) is rapid and accurate. The use of a quartz combustion-tube instead of one of Jena glass is advantageous, since quartz does not retain sulphuric acid, and it is unnecessary to rinse out the tube after each experiment. A tube 50 cm. long and 16 mm. in bore is employed, and as a contact material the platinum star recommended by Dennstedt is quite satisfactory; it is placed in the middle of the tube, and heated to redness by a Teclu burner placed underneath. The products of combustion are absorbed by 4 grms. of pure calcined sodium carbonate contained in two boats, the one nearest the platinum star being 15 c.m. long, and the other of the usual dimensions. The boat containing the weighed material for analysis is heated in an oven and introduced hot into the tube, lest the presence of moisture might cause decrepitation of the absorption material. The connections to the tube are fitted in corks, and both ends are kept cool by water-circulation through lead piping wound round the tube. A piece of sheet-iron channel is placed under the portion of the tube containing the absorption-boats to distribute the heat; the central portion of the tube is heated with direct flame. The oxygen is dried by sulphuric acid, and the progress of the combustion is indicated by the glow of the pyrites. A bubble-counter containing dilute permanganate is placed at the end of the tube, and serves to indicate the completeness of combustion. Combustion is started by heating one end of the boat containing the pyrites; it then proceeds by itself, and is finished off by heating with increasing strength by the Teclu burner. The sodium carbonate is dissolved in bromine water, and neutralised with hydrochloric acid; the pyrites residue is dissolved in 2 c.c. of hydrochloric acid, the iron is precipitated by ammonia, and the filtrate is added to the solution of the sodium carbonate. The sulphuric acid is precipitated by adding to the solution double the theoretical quantity of barium chloride required for precipitation; this is an important detail. The results are in satisfactory agreement with those afforded by Lunge's method; usually they are very slightly higher than the latter.

J. F. B.

Rapid Method of Estimating Zinc. K. Voigt. (*Zeit. angew. Chem.*, 1911, **24**, 2195-2198.)—The following rapid method of estimating zinc in ores, slags, etc., is shown to give results agreeing within about 0·36 per cent. of the gravimetric results, even in the case of substances rich in iron and poor in zinc: From 1 to 3 grms. of the finely powdered sample are decomposed with about 10 c.c. of nitric acid (sp. gr. 1·2) and 20 to 30 c.c. of hydrochloric acid (sp. gr. 1·19), care being taken to break up any deposit of silica. The mass is then transferred to a 300 c.c. flask, and treated with an excess (50 to 60 c.c.) of strong ammonia, which precipitates silica, iron, aluminium, and lead, but forms sufficient ammonium salts to keep the zinc in solution. After being boiled for a short time the contents of the flask are cooled, made up to the mark with dilute ammonia solution (1 : 3), well shaken, allowed to settle, and filtered. Two portions of 100 c.c. each of the filtrate are collected, one of which may be kept for a control gravimetric estimation if required. In the other portion the manganese is precipated by adding bromine and boiling the liquid, after which about 1 grm. of aluminium is added and the boiling gently continued for some minutes until any copper present has been precipitated. About 5 c.c. of a solution of sodium sulphite (1 : 4) and a few c.c. of ammonia solution are next added, and the liquid once more heated to the boiling-point, and immediately filtered. The filter is washed with a dilute ammoniacal solution of sodium sulphite, and the filtrate and washings treated with dilute hydrochloric acid (1 : 3), an excess of 10 c.c. beyond that required for neutrality being added. Finally, the solution (about 150 c.c. in all) is titrated with a solution of 21·63 grms. of potassium ferrocyanide and 14 grms. of crystalline sodium sulphite per litre, the end-point of the reaction being found by spotting tests with a solution of ammonium molybdate (9 grms. per litre) as indicator. Any yellow coloration due to phosphates may be easily distinguished from the brown tint produced by the excess of ferrocyanide solution. An excess of about 0·25 c.c. of the latter in 150 c.c. of liquid is required to give a sharp end-point, and this amount should therefore be deducted from the number of c.c. consumed. C. A. M.

Separation of Zirconium from Iron and Aluminium, and a New Method for the Analysis of Ferro-zircon. M. Wunder and B. Jeanneret. (*Zeitsch. anal. Chem.*, 1911, **50**, 733-735.)—Zirconium oxide is totally unaffected by fusion with sodium carbonate; hence it may be readily separated from aluminium and chromium. The oxides of iron, aluminium, and zirconium are fused in a platinum crucible with about 6 grms. of sodium carbonate; the melt is taken up with water, and, after the addition of 1 grm. of sodium carbonate, the solution is boiled for a few minutes. The mixture of iron and zirconium oxides is collected on a close filter, and if much aluminium or chromium be present, the filter is incinerated and the fusion repeated. The aluminium is precipitated from the filtrate by the addition of an excess of ammonium nitrate. The precipitate is treated on the filter with hot hydrochloric acid (1 : 1), whereby the iron oxide is completely dissolved and the zirconium oxide is unaffected.

The author describes a rapid method of analysing ferro-zirconium alloys; these alloys contain zirconium, iron and silicon, sometimes also aluminium. The alloy is heated with bromine and hydrochloric acid on the water-bath, whereby the zirconium and iron are dissolved, whilst silica remains. The silica is dehydrated by baking at 110° C., taken up with dilute hydrochloric acid, and filtered off; the zirconium, iron, and aluminium in the filtrate are precipitated by ammonia. The weighed silica is volatilised with hydrofluoric acid, any co-precipitated zirconium oxide being left behind and added to the ammonia precipitate. The mixed oxides are fused with sodium carbonate and separated in the manner described above. The presence of bromine will cause loss of iron by volatilisation of ferric bromide during the dehydration of the silica, unless expelled by boiling with nitric acid before evaporation. J. F. B.

APPARATUS, ETC.

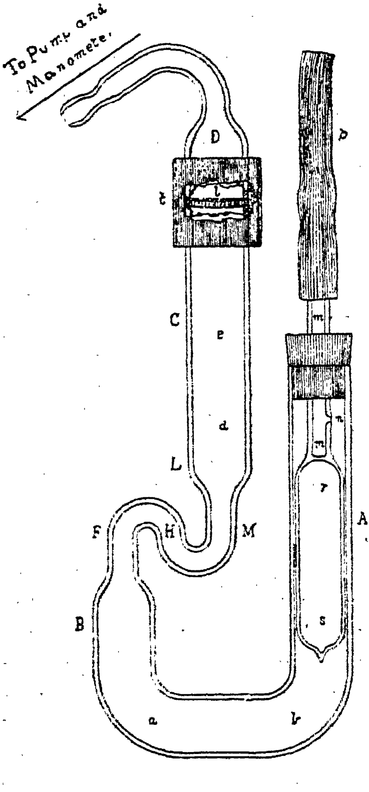
New Method for the Physical Analysis of Soils. J. Dumont. (*Compt. rend.*, 1911, **153**, 889-891.)—The sandy particles of soil separated by simple levigation are generally covered by a humo-argilaceous layer, which in the case of heavy soils may easily lead to false results; this layer may be loosened by cleansing the sand with dilute oxalic acid; further, the slimy and argilaceous matters may be more rapidly and perfectly separated by centrifuging than in the ordinary way. These observations form the basis of a new system of soil analysis. (1) Estimation of the carbonates and cleansing of the fine earth: Two to 5 grms. of the finely powdered dry soil are treated in the calcimeter with an equal weight of oxalic acid for the estimation of carbon dioxide. After the evolution of the gas, the amount of residual acid is calculated, and the liquid is diluted with water to produce a concentration of 2 per cent. of the acid. The earth is boiled with this liquid for half an hour, collected on a filter, extracted with dilute nitric acid, and washed with boiling water. (2) Estimation of humus and separation of the sand: The residue is removed from the filter into a dish, using 75 to 80 c.c. of dilute ammonia, 20 c.c. of ammonia are added, the whole is brought to the boil in order to dissolve the humus, and transferred to a graduated tube. The tube is filled up to a height of 15 cm., shaken, and left at rest for two hours; the liquid is decanted off the sandy layer, the residue is again shaken with dilute ammonia, and the liquid is poured off the coarse sand after settling for five minutes, the fine sand being also collected, dried, and weighed. (3) Separation of the slimy and colloidal components: The liquid from the first decantation is generally coloured by ammonium humate; it contains in suspension the mineral colloids (silicates) and slimy constituents which are to be separated by centrifuging. The liquid is poured into centrifuge tubes closed at the bottom by a small cup in which the deposits are collected, removed, and dried. The time of centrifuging may be varied if it be desired to classify the slimes, but in current practice these are separated as a whole by centrifuging for fifteen minutes at an average speed of 1,000 to 1,200 revolutions per minute. During centrifuging, the colloidal silicates remain in suspension in the ammoniacal solution of humus; they are readily coagulated by ammonium carbonate, and the coagulum is separated by a further centrifuging. These matters are collected and removed in the cups, and the liquid is finally evaporated to dryness on the water-bath, leaving a residue of humus.

J. F. B.

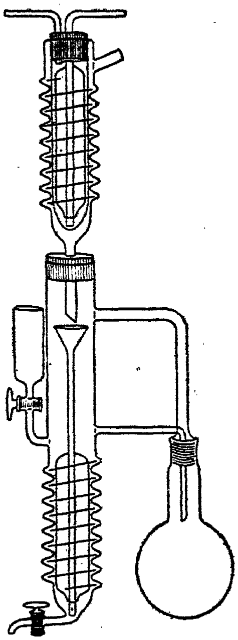
Solubility Estimations in Agricultural Chemistry. A. Rindell. (*Akad. Einladungsch. Helsingfors*, 1910; through *Chem. Zentralbl.*, 1911, II., 1654-1655.)—According to the author's investigations, the usual method for the estimation of citric acid-soluble phosphoric acid in Thomas meal cannot afford a determination of a definite solubility nor show the percentage of phosphoric acid of the supposed solubility. In the case of phosphate fertilisers, the author thinks that the solubility of the phosphoric acid should be determined with a much weaker solvent than the usual 2 per cent. solution of citric acid. In pure water the phosphoric acid of most crude phosphates is considerably less soluble than that of Thomas meal. In the case of potash fertilisers, the solubility, and the acid with which the base is combined are of little importance with regard to the action of the potash. Similar remarks apply to the superphosphates. In the case of precipitated phosphates, estimations of the solubility are of importance. The solubility of the dicalcium phosphate crystallising with 2 mols. of water is about 180 mgrms. of P_2O_5 per litre. In the case of tricalcium phosphate, not too strongly dried, the solubility is about two-thirds of this value; for incinerated tricalcium phosphate the solubility is barely 5 mgrms. of P_2O_5 per litre. The first value is about ten times the concentration of the phosphoric acid solution assimilated by an average crop of barley. In the case of nitrogenous fertilisers, the estimation of solubility is of doubtful importance. For the estimation of assimilable plant-nutrients a solvent of suitable strength must be selected, and used in such a manner in the case of each nutrient that first the corresponding solubility and secondly the quantity of any compounds which may be present having a different solubility are determined. Further, the author has shown that the presence of various compounds of the same plant-nutrient must be indicated by changes in the course of the solubility curve, with concentrations as ordinates and quantities of soil per litre of the solvent as abscissæ, and that the co-ordinates of these change-points have an important meaning, in that each ordinate expresses the solubility of a definite compound, the quantity of which in the soil is shown by the relation of the ordinates to their corresponding abscissæ. J. F. B.

Modified Explosion Eudiometer. F. H. Campbell. (*Chem. News*, 1911, **104**, 235-236.)—The modification consists in the addition of a three-way capillary-bore tap at the head of the eudiometer, by means of which the tube can be filled with mercury when the tap is open to the air. Hydrogen, etc., is passed in with the tap turned so as to communicate with the connection-tubes only, thus sweeping out air, and then the tap being turned again the gas passes into the eudiometer-tube to the desired volume. Another turn of the tap then allows of the admission of the second gas. A. R. T.

Simple Apparatus for Sublimation in a Vacuum. H. Christopher. (*Proc. Chem. Soc.*, 1911, **27**, 236-237.)—Owing to the density of the vapours of some substances, their diffusion to the cool part of a sublimation tube is a very slow process, and the accompanying figure shows the author's device for overcoming the difficulty. The apparatus consists of a tube, ABCD, about 7 inches in height and $\frac{5}{8}$ inch external diameter. The portion *ab* of the tube contains the substance to be sublimed, and the apparatus is heated, immersed to the level of L, in an oil or metal bath, the part C serving as the cooling chamber into which the vapours are carried by a current of air. The constricted portion of the tube, FHM, avoids the falling back of the sublimate in *de* on to the unsublimed substance. The current of air, regulated by a screw clip at P, enters the apparatus through the orifice *n* in the tube *mm*, and passes down the space between the outer wall of the sealed and exhausted tube *rs*, and the inner wall of the sublimation tube A over the substance to the condensing limb C and through a filter *l* to the pump. The filter consists of a disc of filter-paper held against the under side of a perforated porcelain disc, and serves to prevent the blocking of the outlet-tube. The filter rests on the end of the condensing limb and is held in place by the head D and the thick rubber connection *t*. Vortices are formed in the sublimation tube if *rs* be omitted, and some of the vapours carried into A. The working pressure (internal) should be about 40 mm., using a water-jet pump. A. R. T.



New Extraction Apparatus. F. Friedrichs. (*Zeitsch. anal. Chem.*, 1911, **50**, 756.)—An apparatus (see Fig.) is described for the extraction of liquids with ether, the principle of helical contact-surfaces being employed. The ether is vaporised in the round distillation flask, which is attached by a ground-glass joint. The vapour travels up the wide tube into the extraction vessel and is condensed in the helical surface-condenser, the liquid ether then falls into the funnel and is conducted by the central tube to the bottom of the liquid to be extracted. Thence it rises in small drops following the convolutions of the helix to the surface of the liquid. When the layer of ether has risen to the level of the lower horizontal branch it flows back through this tube into the boiling-flask, and the extraction becomes continuous. By means of the tap at the bottom and the tapped funnel, the liquid may be removed and replaced by fresh liquid without interrupting the distillation. J. F. B.



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REVIEWS.

Alcools. Alcool, Alcool Dénaturé, Dénaturants. Par M. Louis Calvet. Librairie Polytechnique Ch. Béranger, Éditeur. 1911. Paris and Liège. Price 5 francs.

This small work is concerned with the analysis of industrial alcohols and of the materials used in denaturation, the more interesting subject of the potable spirit obtained by the distillation of fermented liquor having been dealt with in the same series by M. X. Rocques (*Eaux-de-vie*). The first chapter deals with the sources from which alcohol for industrial purposes is obtained, the methods used for rendering such spirit unfit for use as a beverage are briefly touched on, and mention is made of the ways in which evasion of fiscal regulations is attempted.

A résumé of French and foreign legislation relating to the extraction of revenue from the distilling industry is given in the second chapter, which extends to 76 pages, whilst the third chapter of 94 pages is devoted to the methods of analysis adopted in the case of industrial spirit both for determining the alcohol and detecting and estimating the various additions made to it in compliance with legal requirements.

Since alcohol has to be frequently determined in various products, 12 pages are allotted to the methods which are used in determining the alcohol in spirit of camphor, fruit essences, soaps, etc., and to the estimation of butyl and amyl alcohols in alcoholic liquids. Remembering the divergence of opinion which obtained some years ago as to methods by which the higher alcohols could be most conveniently and accurately estimated, it is rather interesting to find a French book recommending the process of extracting the higher alcohols and oxidising them to the corresponding higher acids—in fact, adopting essentially the process associated with the names of Allen, Marquardt, Schidrowitz, and Mann. The actual modification recommended is that due to Lasserre, the extraction being effected by carbon disulphide instead of carbon tetrachloride.

Further chapters deal with unofficial methods for the detection and estimation of methyl alcohol in presence of ethyl alcohol, and methods of analysis presented officially in other countries than France.

The second part of the book is largely occupied with alcoholometric tables; whilst in the short third section a digest of French legislation concerning alcohol for consumption is given, and also tables of production of alcohol in France since 1840.

The author has done his work carefully, and much information of use to the analyst may be obtained from the work. J. T. HEWITT.

"MATIERES TANNANTS CUIRS." Par L. JACOMET. Librarie Polytechnique Ch. Béranger, Éditeur. 1911. Paris and Liège. Price 5 francs.

This volume is issued under the auspices of the chief chemist of the Ministries of Finance, Commerce, and Agriculture of the French Government, and is one of a number of approved textbooks intended primarily for the use of those chemists engaged in the French Government Laboratories in the examination of the various materials which may come before them from the Customs or other Government departments.

The author has divided this work into six parts:

Part I. deals with the tanning materials, with the qualitative reactions, the various approved methods of analysis and estimation. It gives a list of the common tanning materials, their sources, the average strength, and touches lightly on the various adulterants found in some tanning extracts.

Part II. deals with the hides and skins, methods of analysis, and physical tests.

Part III. deals with gelatine, glue and size, various chemical and physical tests. Short reference is also made to fish-glue, photographic gelatine, and vegetable substitutes.

Part IV. deals with vegetable and animal blacks.

Part V. deals with varnishes, lacquers, and enamels.

Part VI. is a collection of tables giving the qualitative tests for all the different tannins, alone and in admixture, together with useful tables appertaining to this subject.

In Part VII. the author gives a very useful résumé of the various requirements of the French Government with regard to leathers and the various allied products, and the standard of purity, etc., which they are bound to possess to satisfy the Government requirements.

The author also refers briefly to the regulations insisted upon in factories working on Government contracts.

The volume is well illustrated, and forms a useful laboratory handbook for those engaged in the chemistry connected with the leather and allied industries.

J. GORDON PARKER.

FEBRUARY, 1912. Vol. XXXVII., No. 431.

THE ANALYST.

OBITUARY.

We deeply regret to have to record the death of one of the Founders and Past Presidents of the Society of Public Analysts, Dr. John Muter. He died on December 11, 1911, at Worthing. An obituary notice will appear in the next number of this Journal.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE ESTIMATION OF FURFURAL BY MEANS OF FEHLING'S SOLUTION.*

By LEWIS EYNON, B.Sc., F.I.C., and J. HENRY LANE, B.Sc., F.I.C.

(*Read at the Meeting, December* 6, 1911.)

According to J. T. Flohil, under certain conditions Fehling's solution is reduced by furfural in direct proportion to the quantity of the latter, and he has proposed a method for the determination of pentosans in which, after distilling the material with acid, the furfural in the distillate is determined by means of Fehling's solution (*Chem. Weekblad.*, 1910, **7**, 1057; Analyst, 1911, **36**, 161).

The method is conducted as follows: Fifty c.c. of the acid distillate (the total quantity of which amounts to 400 c.c.) are neutralised, treated with 20 c.c. of Fehling's solution, and made up to 100 c.c., the whole being boiled for thirty-five minutes under a reflux Liebig's condenser. In order to avoid any loss of furfural, a few pieces of ice are placed in the upper end of the condenser-tube, which is funnel-shaped. A blank determination must be made in which the same quantity of sodium chloride is present. Flohil finds that under these conditions the furfural-copper ratio is 0·3775 : 1—*i.e.*, that 1 molecule of furfural is equivalent to 4 atoms of copper or 2 atoms of oxygen. The amount of cuprous oxide may be found, not only by direct weighing, but indirectly by the iodometric determination of the unreduced copper (Schoorl, *Z. Angew. Chem.*, 1899, **12**, 633).

We have carried out series of experiments to ascertain the influence of variations in the concentrations both of furfural and of sodium chloride, and to determine the degree of accuracy that can be attained by Flohil's method. We find that, in heating the furfural solution with Fehling's solution under an ordinary Liebig's condenser, the use of ice to insure the complete condensation of the furfural is quite unnecessary. Contrary to Flohil's statement, we find that the copper-reducing power of furfural is not independent of the concentration, the furfural-copper ratio ranging from 0·376 to 0·450, as the amount of furfural present is increased from 0·01 to 0·05 grm. There appears to be no ground for Flohil's assumption of a simple stochiometric relation between the furfural and the copper.

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* This work was carried out in accordance with the terms of the Analytical Investigation Scheme.

The furfural employed was purified by distillation in steam; it was then dried over calcium chloride and redistilled. We have found that the reducing power of dilute aqueous solutions of furfural undergoes no change in the course of several weeks. The Fehling's solution used was prepared by mixing equal volumes of a solution of 69·26 grms. of crystallised copper sulphate per litre, and a solution containing 100 grms. of sodium hydroxide and 348 grms. of Rochelle salt per litre.

Method of Procedure.—A solution containing not more than 0·05 grm. of furfural was mixed with 20 c.c. of Fehling's solution and a known quantity of a salt solution in a flask of about 200 c.c. capacity, the whole being made up to 100 c.c. with water. The flask was fitted with a vertical reflux condenser, and heated on an asbestos card over a flame of such size that the liquid was brought to the boiling-point in twelve to fifteen minutes, after which it was kept boiling for exactly thirty-five minutes. The precipitate of cuprous oxide was then collected in a Soxhlet tube, washed with water and alcohol, dried at 110° to 115° C. and weighed, after which it was reduced to copper and weighed again.

Table I. gives the results of a number of blank determinations in which mixtures of 20 c.c. of Fehling's solution, various quantities of salt, and sufficient water to make up 100 c.c., were heated as above described.

TABLE I.

Weight of Sodium Chloride: Grms.	Cuprous Oxide: Mgrms.	Copper: Mgrms.
0·0	5·5	4·9
5·6	9·3	8·3
8·0	10·5	9·3
10·4	11·7	10·4

Table II. shows the reducing power of furfural at various concentrations and in absence of salt. The weights of cuprous oxide given in column 2 are those found less 5·5 mgrms., the correction required under these conditions (see Table I.). The weights of copper given in column 3 (*a*) are those found less 4·9 mgrms., the correction required. The weights of copper given in column 3 (*b*) are calculated from the weights of cuprous oxide given in column 2. The close agreement between the figures in column 3 (*a*) and (*b*) shows that there is no necessity to reduce the cuprous oxide to metallic copper. The reducing power of the furfural is not independent of the concentration, the furfural-copper ratio increasing from 0·362 to

0·452, as shown in column 4. This may be the result of mass action; in presence of 0·05 grm. of furfural, the greater part of the copper in 20 c.c. of Fehling's solution is reduced. In addition to the results given in the table, fourteen other determinations were made with 0·05 grm. of furfural (the maximum quantity employed). In most cases the results agreed well with those given in the table, but in five cases differences of as much as 4 to 6 mgrms. in the weight of cuprous oxide (*i.e.*, 3 to 5 per cent.) were found.

TABLE II.

1.	2.	3.		4.
Weight of Furfural employed: Grm.	Weight of Cuprous Oxide (corrected): Mgrms.	Weight of Copper (corrected)— (*a*) Mgrms. found.	Weight of Copper (corrected)— (*b*) Mgrms. calculated.	Ratio $\frac{\text{Furfural}}{\text{Copper}}$.
0·0100	30·8 31·8	27·1 28·0	27·3 28·2	0·362
0·0200	57·2 54·8	50·8 48·6	50·8 48·7	0·402
0·0300	80·5 81·3	72·1 72·1	71·5 72·2	0·417
0·0400	101·7 101·4	90·1 89·7	90·3 90·0	0·444
0·0500	126·0 123·7	111·6 109·6	111·9 109·8	0·452

In the well-known process of determining pentosans which involves distilling the material with 12 per cent. hydrochloric acid, a distillate containing about 10 per cent. of the acid is obtained. Since, according to Flohil's method of copper reduction, 50 c.c. of the distillate are used, we have determined the reducing power of furfural at various concentrations, in presence of a quantity of sodium chroide (8 grms.), equal to that obtained by neutralising 50 c.c. of 10 per cent. hydrochloric acid with sodium carbonate.

Table III. shows the reducing power of furfural at various concentrations in presence of 8 grms. of sodium chloride. The weights of cuprous oxide given in column 2 are those found less 10·5 mgrms., the correction required under these conditions (see Table I.). The weights of copper given in column 3 (*a*) are those found less 9·3 mgrms., the correction required. The weights of copper given in column 3 (*b*) are calculated from the weights of cuprous oxide given in column 2. Here again the reducing power of the furfural is not independent of the concentration.

TABLE III.

1.	2.	3.		4.
Weight of Furfural employed: Grm.	Weight of Cuprous Oxide (corrected): Mgrms.	Weight of Copper (corrected)— (a) Mgrms. found.	Weight of Copper (corrected)— (b) Mgrms. calculated.	Ratio $\frac{\text{Furrural}}{\text{Copper}}$.
0·0100	29·3	25·6	26·0	0·399
	28·7	25·4	25·5	
	27·0	23·6	24·0	
0·0200	58·1	50·9	51·6	0·394
	56·3	49·2	50·0	
	58·7	52·1	51·0	
0·0300	82·5	72·5	73·3	0·418
	81·9	72·0	72·7	
	80·7	71·4	71·7	
0·0400	103·4	91·6	91·8	0·429
	106·9	95·4	94·9	
	104·8	93·0	92·1	
0·0500	125·9	—	111·8	0·445
	127·2	112·9	113·9	
	126·5	110·7	112·3	

It is evident from a comparison of Tables II. and III. that the reducing power of the furfural *per se* is independent of the presence of salt—*i.e.*, the weight of cuprous oxide obtained by the action of a given weight of furfural on Fehling's solution in presence or absence of salt, less the weight of cuprous oxide obtained in a blank experiment with the same quantity of sodium chloride, is constant. The values given in Tables II. and III. being, therefore, practically the same, they have been employed in plotting the curve shown below; the points indicated by the small circles represent the mean weights of cuprous oxide from Table II., whilst those indicated by the crosses represent the mean weights of cuprous oxide from Table III. The values given in Table IV. have been taken from the curve. By means of Tables I. and IV. the quantity of furfural corresponding to the weight of cuprous oxide obtained by the above-described method may be found. We have confirmed Flohil's statement that the unreduced copper in the liquid after boiling may be determined by Schoorl's iodometric method (*loc. cit.*). For this purpose the contents of the flask are cooled in running water, treated with 10 c.c. of a 20 per cent. solution of potassium iodide and 10 c.c. of a 25 per cent. solution of sulphuric acid

(1·5 parts of concentrated acid to 8·5 parts of water by volume), and then titrated with $\frac{N}{10}$ thiosulphate solution.

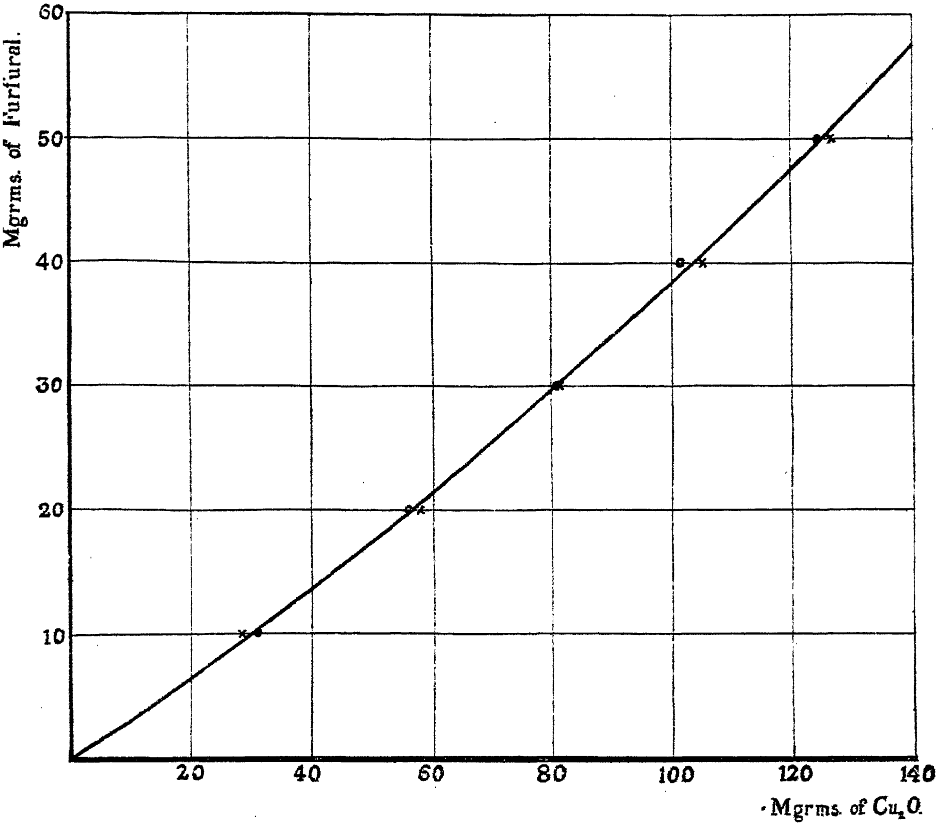


TABLE IV.

Weight of Furfural : Grm.	Weight of Cuprous Oxide (corrected) : Mgrms.	Weight of Copper (corrected) : Mgrms.	Ratio $\frac{\text{Furfural}}{\text{Copper}}$.
0·0100	30·0	26·6	0·376
0·0200	57·0	50·6	0·395
0·0300	81·0	71·9	0·417
0·0400	103·5	91·9	0·435
0·0500	125·0	111·0	0·450

DISCUSSION.

The CHAIRMAN (Mr. LING) remarked that the estimation of furfural was of great importance in the estimation of pentoses and pentosans, and two methods had hitherto been available, the furfural being estimated in the one case as phenyl hydrazide, and in the other as phloroglucide. Both of these methods emanated from Professor Tollens. The estimation as phenyl hydrazide was very uncertain in its results, and the phloroglucide method was more satisfactory, but phloroglucinol was a somewhat expensive substance. He had tried the copper-reduction method some years ago, but had not been successful with it. He had applied it in what seemed to him to be a rational manner, by adding an excess of Fehling's solution and heating the mixture in a closed vessel for a certain time in the water-bath. About twelve months ago, however, Flohil had found it possible to obtain much better results by using a reflux condenser, and it was at his (Mr. Ling's) suggestion that the present authors undertook the revision of Flohil's results under the Society's Research Scheme; and having seen their experiments and read their paper, he must say that he considered their investigation to be an ideal one from the point of view of the Research Scheme. Such a method had been much needed, and, as the result of Messrs. Eynon and Lane's work, it was rendered quite as satisfactory as the phloroglucinol method, whilst it had the additional advantage of being more expeditious.

Mr. A. E. PARKES asked whether it would be possible to apply this method to the determination of furfural in spirits.

The CHAIRMAN observed that, the method being a reduction method, could not be used in the case of spirits, which contained other reducing substances as well as furfural.

Dr. SCHIDROWITZ remarked that some ten years ago Dr. Hewitt had tried a method of this kind for the estimation of aldehydes in spirits, but had found it to be inapplicable owing to the smallness of the quantities of aldehydes that were present.

Mr. A. CHASTON CHAPMAN asked at what degrees of furfural concentration the respective ratios of 0·376 : 1 and 0·450 : 1 were obtained.

Mr. W. BACON asked what were the limits of concentration most applicable to this process.

Mr. EYNON, in reply, said that the ratio of 0·376 : 1 was obtained at a furfural concentration of 0·01 grm. in the reaction mixture, and that of 0·450 : 1 at a concentration of 0·05 grm. The quantity of furfural present must be within the limits of 0·01 and 0·05 grm., unless a larger quantity of Fehling's solution be taken, and then, of course, the tables they had given would not apply.

* * * * *

COMPOSITION OF AUSTRALIAN (VICTORIAN) MILK.

By E. HOLL MILLER.

(*Read at the Meeting, December* 6, 1911.)

The average composition of 11,140 samples of milk from farms, analysed in the Anglo-Australian Milk-Preserving Company's laboratory during the year September, 1909, to August, 1910, inclusive, is given in the following table. The period chosen is one complete year; spring embraces the months September to November, summer December to February, autumn March to May, and winter June to August.

	Morning Milk.				Evening Milk.				Mean.				Approximate Corresponding English Month.
	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	
1909.		%	%	%		%	%	%		%	%	%	
September	1·0325	12·94	3·90	9·04	1·0328	13·33	4·16	9·17	1·0326	13·13	4·03	9·10	February.
October ...	1·0319	12·74	3·86	8·88	1·0326	13·07	3·99	9·08	1·0322	12·90	3·92	8·98	March.
November	1·0315	12·71	3·92	8·79	1·0320	12·90	3·97	8·93	1·0317	12·80	3·94	8·86	April.
December	1·0298	12·40	4·01	8·39	1·0302	12·58	4·08	8·50	1·0300	12·49	4·04	8·44	May.
1910.													
January ...	1·0270	11·71	4·02	7·69	1·0270	11·72	4·03	7·69	1·0270	11·71	4·02	7·69	June.
February	1·0281	12·02	4·05	7·97	1·0283	12·03	4·02	8·01	1·0282	12·02	4·03	7·99	July.
March ...	1·0305	12·74	4·15	8·59	1·0307	12·88	4·23	8·65	1·0306	12·81	4·19	8·62	August.
April ...	1·0306	13·14	4·46	8·68	1·0307	13·46	4·71	8·75	1·0306	13·30	4·58	8·72	September.
May ...	1·0303	13·02	4·43	8·59	1·0308	13·37	4·61	8·76	1·0305	13·19	4·52	8·66	October.
June ...	1·0309	12·84	4·15	8·69	1·0314	13·28	4·41	8·87	1·0311	13·06	4·28	8·78	November.
July ...	1·0312	12·74	4·00	8·74	1·0318	13·31	4·35	8·96	1·0315	12·90	4·17	8·73	December.
August ...	1·0303	12·39	3·90	8·49	1·0310	12·97	4·24	8·73	1·0306	12·68	4·07	8·61	January.
Average ...	1·0304	12·62	4·07	8·55	1·0310	12·96	4·23	8·70	1·0307	12·79	4·15	8·62	

It will be noticed that the poorest milk occurs in the month of January, which month approximates to the English month of June. The average difference between morning and evening fat is 0·16 per cent. This figure is lower than that given by Richmond for English milk—viz., 0·35 per cent.

It will be noticed that the evening milk is the richer in fat, while the sp. gr. of the morning's milk is lower than that of the evening's milk. With English milk the sp. gr. of the morning's milk is generally higher than that of the evening's milk.

On comparing the mean average composition with that given by Richmond ("Dairy Chemistry") for English milk, the sp. gr. is found to be lower, and the fat higher.

The lowness of sp. gr. for the months of December, January, and February, is ascribed to a flush of green herbage caused by copious rains.

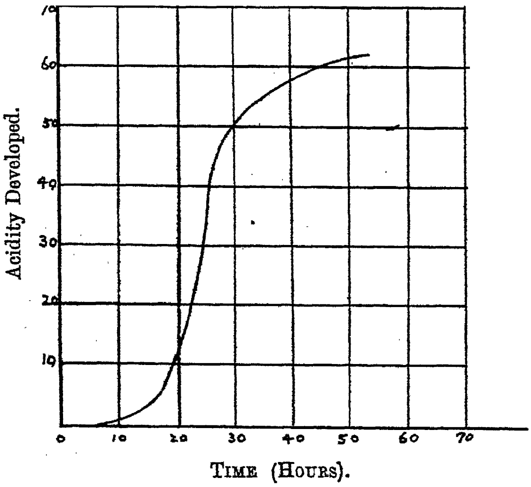
Aldehyde Figure.—The aldehyde figure of a considerable number of the above samples has been found to average 18·5° using $\frac{N}{10}$ strontia, and 17° using $\frac{N}{10}$ soda.

This is a ratio of approximately 1·1, and corresponds with the results of Richmond and Miller (ANALYST, 1906, **31**, 224).

The ratio $\frac{\text{protein}}{\text{aldehyde}}$ has been found to be 0·173 (using strontia), the figure given by Richmond (ANALYST, 1911, **36**, 9) being 0·170.

It was found that milk curdles on boiling when it had developed an acidity of 13° above its normal acidity. This figure is in accordance with that given by Harrison and Richmond (ANALYST, 1900, **25**, 116).

The following curve graphically illustrates the souring of milk:



Time.	*Acidity.*
6	0
16	4
23	20
25	33
27	49
31	50
33	55
50	61
53	63

It will be noticed that there is the usual break in the curve, occurring at 45° and corresponding to the acidity at which the micro-organisms cease to increase.

Acidity.—The average normal acidity was found to be 18°.

Ash.—The percentage of ash determined from a considerable number of samples was found to average 0·72 per cent.

Cl, CaO, and P_2O_5.—The average percentage of these constituents was found to be as follows: Cl, 0·08 per cent.; CaO, 0·18 per cent.; P_2O_5, 0·21 per cent.

A sample of tinned sterilised milk of German origin, imported into Java, gave the following results on analysis:

Sp. gr.	1·0325	Acidity	23°
Total solids	12·21 per cent.	Aldehyde figure ...	21° (strontia)
Fat (Gottleib)	3·21 ,,	Ratio $\frac{\text{protein}}{\text{aldehyde}}$ ...	0·171
Ash	0·72 ,,		
Solids-not-fat	9·00 ,,	Chlorine	0·090
Protein (N × 6·38) ...	3·60 ,,	CaO	0·160
Lactose	4·68 ,,	P_2O_5	0·230

COMPOSITION OF AUSTRALIAN SWEETENED CONDENSED MILK.

By E. HOLL MILLER.

(*Read at the Meeting, December* 6, 1911.)

The following two tables give the composition of samples of five different brands of sweetened condensed milk purchased in Victoria, and six purchased in New South Wales.

These figures are of some interest, as all the milks were manufactured in Australia.

TABLE I.

Purchased in Victoria.

	Brand No. 1.	Brand No. 2.	Brand No. 3.	Brand No. 4.		Brand No. 5.		
				Sample *a*.	Sample *b*.	Sample *a*.	Sample *b*.	Sample *c*.
Total solids ...	72·18	72·96	73·740	76·80	76·24	75·00	74·99	74·74
Fat	10·03	11·41	10·800	10·50	10·63	10·11	10·27	9·61
Ash	1·91	1·90	1·870	2·04	2·01	1·93	1·90	1·94
Solids-not-fat ...	62·15	61·55	61·940	66·30	65·61	64·89	64·72	65·13
Protein (N × 6·38)	9·68	9·20	9·300	9·69	10·12	9·69	9·00	9·05
Total sugars ...	50·56	50·45	50·700	54·59	53·48	53·27	53·82	54·14
Chlorine ...	0·19	0·18	0·188	0·18	0·24	0·23	0·22	0·23
Acidity	43·8°	51°	46·9°	48·6°	49·4°	46·5°	45·5°	45·6°

TABLE II.

Purchased in New South Wales.

	Brand No. 1.	Brand No. 2.	Brand No. 3.	Brand No. 4.	Brand No. 5.	Brand No. 6.
Total solids ...	75·87	67·53	76·14	74·77	71·38	75·87
Fat	10·68	9·41	9·28	9·81	9·93	9·05
Ash	2·10	1·82	2·11	1·82	1·84	2·10
Solids-not-fat ...	65·19	58·12	66·86	64·96	61·45	66·82
Protein (N × 6·38)...	10·25	9·14	9·84	8·95	9·39	9·10
Total sugars ...	52·84	47·16	59·91	54·19	50·22	55·62
Chlorine	0·36	0·20	0·21	0·19	0·19	0·24
Acidity	51°	56·8°	51·84°	44·64°	50·05°	49·75°

For analysis the whole of the contents of the tin were turned out into a basin and thoroughly mixed, and the portions required were taken from the mixed sample.

The fat was estimated by Richmond's modification of the Rose-Gottleib method. Other constituents were estimated in the usual manner.

Tyrosin was detected in all the above samples with the exception of Nos. 1 and 3, Table I.

I have found that tyrosin makes its appearance in sweetened condensed milk between the end of the third and fourth month after manufacture. Samples six months old show distinct crystalline tufts of tyrosin, which can generally be detected on the first slide prepared, when examined under the microscope with a one-sixth inch objective.

Samples twelve months old show fairly large round crystalline feathery tufts. These tufts of crystals all approximate to the same size in the same tin of milk, and the size and number of them form a fairly good criterion of the age of the sample.

* * * * *

THE ALDEHYDE FIGURE OF BUTTER.

By E. HOLL MILLER.

(*Read at the Meeting, December* 6, 1911.)

It has been pointed out by Steinegger that formaldehyde combines with milk proteins to form additive compounds, probably of the type $\mathrm{R}<\begin{matrix}\mathrm{NCH_2}\\ \mathrm{COOH}\end{matrix}$, with an increase in the acidity of the milk, which is proportional to the total nitrogen present. Steinegger's method has been modified by Richmond and Miller (chiefly by substituting the use of strontia for soda), who have shown that under their conditions the method is a rapid and approximately accurate one for estimating the percentage of proteins in milk; and it has been further shown by Richmond (Analyst, 1911, **36**, 9) that the degree of accuracy of the determination is of the same order as the Gerber method for the estimation of fat.

A number of experiments have been carried out in order to determine whether use could be made of this method to estimate with a fair degree of accuracy the proteins in butter, as it was recognised that a rapid method for such would be of considerable value in butter-factory routine analysis.

The following procedure was found to be the best for carrying out the determination: Approximately 10 grms. of butter are weighed into a tared beaker, which is placed in a water-bath at 60° to 70° C., until the butter is completely melted. Twenty-five c.c. of water at about 65° C. are added, and followed by 1 c.c. of a 0·5 per cent. solution of phenolphthaleïn. The contents are well agitated. Approximately $\frac{N}{20}$ alkali is then run in until a faint permanent pink tint is formed. If it is found that the end point is masked by the yellow colour of the butter-fat, the contents of the beaker should be allowed to settle, and the bottom aqueous layer observed, and the addition of alkali continued until the pink tint is obtained. Five c.c. of strong formaldehyde solution are next added, and the contents of the beaker well agitated; $\frac{N}{20}$ strontia is then run in until the pink tint is again produced in the aqueous portion.

The number of c.c. of $\frac{N}{20}$ alkali used in the second titration, less the amount equivalent to the acidity of the formaldehyde solution added, is proportional to the protein present. One c.c. of $\frac{N}{20}$ strontia is equivalent to 0·01355 grm. of protein nitrogen, assuming the proportion of casein to albumin to be 7 to 1 (Richmond and Miller, ANALYST, 1906, **31**, 224).

Thus, when 10 grms. of butter are taken, the number of degrees of acidity produced when multiplied by 0·170 gives the percentage of protein.

The following are some of the results obtained:

SALTED BUTTER ONE MONTH OLD, PREPARED FROM RIPENED CREAM.

Aldehyde Figure, °.	rotein (Kjeldahl, × 6·38) per Cent.	Ratio $\frac{\text{Protein}}{\text{Aldehyde.}}$	Protein calculated, ° Aldehyde × 0·170.	Difference.
3·50	0·620	0·177	0·595	−0·025
3·00	0·495	0·165	0·510	+0·015
4·00	0·690	0·172	0·680	−0·010
3·50	0·600	0·171	0·595	−0·005
5·00	0·860	0·172	0·850	−0·010
4·13	0·720	0·174	0·700	+0·020

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Water in Butter. A. C. D. Rivett. (*Chem. News*, 1911, **104**, 261-263.)—The author employs the calcium carbide method, and determines the loss of acetylene by weighing, thus arriving at the amount of water present in the sample. A special form of apparatus is employed, in which a known weight of the butter in a lower chamber can be heated, in the weighed apparatus, after allowing it to become mixed with a quantity of calcium carbide placed in an adjacent chamber. The acetylene passes through a tower of glass-wool mixed with carbide, which traps any moisture, and is finally swept out of the apparatus by means of a current of dried air. Experiments show that 0·696 grm. of acetylene represents 1 grm. of water (theory = 0·722 grm. C_2H_2), and the calculation is made on this basis. The butter and carbide mixture should be heated with the direct flame to a temperature similar to that ordinarily reached in the direct heating method. The results are usually 0·2 to 0·3 per cent. lower than by the last-named method, in which probably volatile matter other than water-vapour escapes during heating. The method of heating butter with alcohol at 100° C. to expel the water gives results about 0·1 per cent. higher than the carbide method. A. R. T.

Influence of Certain Preservatives on the Keeping Properties and Composition of Butter. K. Fischer and O. Greunert. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 553-582.)—Parallel experiments carried out on the effect of various substances on the preservation of butter and margarine showed that benzoic acid, salicylic acid, and boric acid, failed to prevent the butter and margarine from becoming rancid after the lapse of a short time, even when added in quantities up to 1 per cent. On the other hand, butters and margarine containing 3 per cent. of common salt remained fresh as regards taste and odour for from two to three months. The changes taking place in the butter and margarine containing the first-mentioned substances were due to decomposition of the glycerides and the proteins; this decomposition was not decreased proportionally to the quantities of the preservative substances added. W. P. S.

Estimation of Cocoa-Husks in Cocoa. C. Ulrich. (*Arch. Pharm.*, 1911, **249**, 524-597.)—Analyses of many samples of the different kinds of cocoa upon the market are given, and the methods of estimating cocoa-husk are critically examined (*cf.* ANALYST, 1909, **34**, 134). The iodine value of the fat of the roasted beans examined ranged from 32·20 to 35·6 (mean, 34·1), whilst in the case of unroasted beans the limits were 34·7 to 36·5 (mean, 35·5). Prochnow (*Dissertation*) confirmed the results of Welmans (*Z. öffent. Chem.*, 1901, **7**, 500), that the fat from the shells had a considerably higher iodine value (43·2 to 46·2 for six samples) than the bean fat; but the author has obtained lower values than these—34·8 to 37·9 (mean, 36·3) for the fat from roasted shells, and 36 to 38 (mean, 37·3) for the fat from unroasted shells. Hence it is not possible to detect the presence of even 30 per cent. of husks, as claimed by Welmans, by means of the increase in the iodine value of the fat. With regard to the elutriation method of separating the husk, devised by Filsinger and Drawe (ANALYST, 1903, **28**, 216), the author's experiments indicate that as little as 7·5 per cent. of added husk may be detected and estimated in normal cocoa (30 per cent. of fat and moisture), provided that the husk is present in a very finely powdered condition, and that the factor (for the loss) of 1·43 proposed by Drawe be increased to 1·72. Estimation of the amount of crude fibre by the method of König (ANALYST, 1898, **23**, 47), and the modification of Matthes and Müller (ANALYST, 1906, **31**, 362), gave the following results with eight different kinds of cocoa: Roasted kernels 5·79 to 7·9, and roasted husks 13·5 to 17·7 per cent., calculated on the fat-free substance. The method would not enable a smaller quantity than 27·5 per cent. of husks to be detected. In like manner, estimation of the pentosans (see Hehner and Skertchley, ANALYST, 1899, **24**, 178) is only of value in the case of mixtures containing more than 25 per cent. of husk. The following new method, based on the precipitation of cocoa-red by means of ferric chloride from an acetic acid extract of the cocoa, is claimed to detect with certainty the addition of 10 per cent. and upwards of cocoa-husks to a normal cocoa: One grm. of the finely-powdered, anhydrous, fat-free sample is boiled for three hours beneath a reflux condenser with 120 c.c. of pure 50 per cent. acetic acid, after which the contents of the flask are cooled, made up to 150 c.c., well shaken, and allowed to stand for at least twelve hours. The liquid is then filtered, and 135 c.c. of the filtrate are treated with 5 c.c. of concentrated hydrochloric acid and 20 c.c. of 20 per cent. ferric chloride solution, then boiled for ten minutes under a reflux condenser, rapidly cooled, and transferred to a beaker. After standing for at least six hours, the precipitate is collected upon a dried tared filter, washed until the washings are free from iron, dried for six hours at 105° C., and weighed. The following results, calculated upon the anhydrous and fat-free cocoa-beans and upon the original air-dried material, were thus obtained:

Cocoa.	Roasted Beans.				Raw Beans.			
	Estimations.	Ferric Chloride Precipitate.			Estimations.	Ferric Chloride Precipitate.		
		Extremes.	Mean.	On Original Material.		Extremes.	Mean.	On Original Material.
		Per Cent.	Per Cent.	Per Cent.		Per Cent.	Per Cent.	Per Cent.
Accra	6	13·07—13·69	13·52	6·34	3	14·56—15·07	14·82	6·29
Ariba	18	12·77—13·97	13·50	6·99	3	12·22—12·56	12·82	5·84
Bahia	14	10·71—12·49	11·39	5·47	3	12·13—12·73	12·40	5·59
Ceylon ...	6	13·67—13·89	13·79	6·45	—	—	—	—
Guayaquil ...	12	15·13—16·82	16·11	7·48	3	16·44—17·08	16·67	7·31
Samoa ...	12	13·82—14·84	14·34	6·69	3	14·80—15·0	14·90	6·49
St. Thomé ...	18	13·11—14·27	13·75	6·12	—	—	—	—
Trinidad ...	12	13·51—14·56	14·11	6·14	3	13·11—13·36	13·26	5·91
Mixture of all	7	13·02—13·78	13·51	5·81	1	14·33	14·33	6·33

Extracts of the husks gave no precipitate under the same conditions, whilst mixtures of the different kinds of cocoa with 10 per cent. of husks gave results corresponding within 0·9 per cent. of the calculated smaller amounts of precipitate. Bahia and Guayaquil cocoa thus give results deviating considerably from those of the other kinds of cocoa, but it is pointed out that, in practice, commercial (normal) cocoa usually consists of a mixture of two or more brands, and that in the case of such mixtures the method will readily detect 10 per cent. of husks. If the cocoa is known to be a Bahia product alone, the amount of the precipitate must be as low as 9·68 per cent. before the presence of 10 per cent. of husk is indicated. In like manner, in the case of Guayaquil cocoa it is also necessary to know the brand, since otherwise a precipitate of 13·70 per cent., as obtained from a mixture of that cocoa with 10 per cent. of husks, would indicate a pure sample. Not until as much as 21 per cent. of husks has been added to Guayaquil cocoa does the iron method give a value of 11·28, which in the case of a normal cocoa would indicate the presence of 10 per cent. of husk. C. A. M.

Composition of Honey from Various Countries. **K. Lendrich and F. E. Nottbohm.** (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 633-643.)—The following average results were obtained on the analysis of sixty-three samples of honey imported into Germany, the samples being taken either from the packages in the holds of the vessels or in the Customs warehouses:

Country of Origin.	Water.	Ash.	Sodium Chloride.	Acidity (c.c. $\frac{N}{1}$ Alkali per 100 Grms.)	Invert Sugar.	Sucrose.	Non-Sugars.	Proteins (N×6·25).
	Per Cent.	Per Cent.	Per Cent.		Per Cent.	Per Cent.	Per Cent.	Per Cent.
France (2 samples)	19·81	0·30	—	3·50	75·86	0·15	4·18	0·58
Italy (1 sample) ...	17·20	0·06	0·02	1·80	76·62	3·19	2·99	0·21
Hawaii (8 samples)	16·51	0·53	0·38	0·86	80·18	1·76	1·51	0·17
California (8 samples)... ...	17·37	0·22	—	2·39	74·15	2·46	6·03	0·28
Mexico (4 samples)	19·62	0·19	—	2·50	72·81	1·59	5·97	0·46
Jamaica (9 samples)	19·37	0·30	—	2·69	74·25	1·52	4·85	0·50
Hayti (2 samples)	19·98	0·25	—	2·70	73·58	1·49	4·96	0·39
St. Domingo (5 samples)... ...	18·64	0·45	—	2·08	3·85	1·29	6·25	0·43
Peru (7 samples) ...	19·24	0·39	—	2·86	76·45	1·22	3·08	0·40
Chili (14 samples)	17·84	0·20	—	2·83	76·43	1·41	4·32	0·50
Australia (4 samples)	15·32	0·32	—	1·13	75·47	0·22	6·98	0·28
All samples:								
Minimum ...	14·59	0·05	0·02	0·60	69·60	0·04	0·94	0·12
Maximum ...	21·34	0·63	0·42	5·90	81·56	5·36	8·86	1·07
Average	18·26	0·29	—	2·30	75·42	1·48	4·65	0·38

Only one of the samples yielded a positive reaction with Fiehe's test. This sample contained no diastase, was dark brown in colour, and had evidently been overheated in manufacture. W. P. S.

Bassia Kernels and Fats. (*Bull. Imp. Inst.*, 1911, **9**, 228-236.)—The seeds come into the market under the name of "mowra" and "illipe" seeds. The following table shows the constants of the fats from some authentic samples:

	Bassia Latifolia.		*Bassia Longifolia.*		*Bassia Butyracea.*		*Bassia Longifolia.*
	From Nagpur, India.	Fat prepared in India.	Fat from Madras, prepared in India.	From Madras.	From Gonda, United Provinces.	From Gonda, prepared in India.	Sample of "Mee Oil" as received from Ceylon.
Yield of fat per cent. from kernels...	46	—	—	57·8	61·2	—	—
Sp. gr. at $\frac{100^\circ \text{C.}}{15·5^\circ \text{C.}}$	0·857	0·870	0·864	0·861	0·856	0·862	0·861
Acid value	—	20·0	5·0	19·7	59·6	20·7	25·7
Saponification value...	189·8	194·4	198·2	195·3	200·0	195·3	191·5
Iodine value, per cent.	57·6	76·8	60·0	60·0	39·6	42·7	61·2
Hehner value, per cent.	94·2	86·7	87·4	95·5	96·6	94·6	—
Reichert-Meissl value	0·2	1·0	3·6	2·35	nil	4·3	—
Unsaponifiable matter, per cent. ...	2·0	1·7	2·1	1·4	2·2	2·8	—
Titer test, °C.	43·2	36·0	36·0	45·0	51·5	48·2	40·2

The fat from *B. butyracea* is harder and whiter than that from the other two species, but it is not exported, the available supply being consumed locally. Bassia kernels at present are chiefly crushed in France and Germany, since in England the expressed cake is considered poisonous for cattle. It has been shown (*Bio-Chemical J.*, 1910, 93) that such cake contains a saponin-like glucoside with marked physiological activity when subcutaneously injected, but apparently without action when fed to cattle. H. F. E. H.

Characteristics of Palm Oil from Different Varieties of Palm. A. Hébert. (*Bull. Soc. Chim.*, 1911, [iv.], **9**, 1083-1085.)—The samples of palm oil examined were extracted by means of petroleum spirit from the fruit of palm-trees grown in Dahomey. All the fats are used by the natives as food, with the exception of that obtained from the variety *repanda*, which is reputed to cause headache and sickness. The yields and analytical values were as follows:

FAT OF *Elæis Guineensis.*

	Sub-species *E. Nigrescens.*						Sub-species *E. Virescens.*		Commercial Palm Oil.
	Variety *Communis.*	Variety *Vulgaris.*		Variety *Semper-nigra.*	Variety *Pisifera.*		Variety *Repanda.*	Variety *Gracilinux.*	
Oil from pulp, per cent.	41·0	43·0	47·0	52·0	59·0	60·0	63·0	52·0	—
Oil from whole fruit, per cent.	22·0	21·0	29·0	16·0	42·0	56·0	32·0	30·0	—
Melting-point of fat, ° C. ...	42·0	42·0	43·0	44·0	45·0	44·0	43·0	42·0	—
Sp. gr. at melting-point	0·882	0·881	0·888	0·892	0·891	0·889	0·884	0·889	0·945 at 15° C.
Saponification value	201·0	201·0	196·0	196·0	197·0	210·0	196·0	198·0	202·0
Reichert value ...	0·8	0·8	0·8	0·9	1·3	1·1	0·8	1·1	0·5
Iodine value ...	43·8	48·0	52·2	52·2	50·2	45·6	52·1	55·6	52·0
Hehner value ...	98·0	96·2	96·2	98·1	97·7	97·8	95·0	97·7	95·0
Melting-point of fatty acids, ° C.	46·0	48·0	48·0	48·0	47·0	46·5	44·0	44·0	47·0

C. A. M.

Analysis of Mustard Flour. A. Domergue. (*J. Pharm. Chim.*, 1911 [vii.], **4**, 494-496.)—In estimating the proportion of essential oil in mustard flour it is necessary to prevent the distilling vapours coming into contact with rubber connections. For this purpose the author employs an apparatus in which one end of the condenser is connected with the flask by means of a ground-in stopper, whilst the other end is bent downwards at a right angle into a graduated cylinder. From 60 to 65 c.c. of the distillate are collected as described by Lenormand, after which 20 c.c. of $\frac{N}{10}$ silver nitrate solution are added, the volume made up to 100 c.c. and the estimation completed. The proportions of essential oil most usually found in commercial samples of mustard flour are from 0·2 to 0·9 per cent. The French Codex specifies 0·70 per cent. as the minimum for black mustard seed. C. A. M.

Composition of Capsicum (Cayenne Pepper). A. von Sigmond and M. Vuk. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 599-605.)—Analyses of twenty-five samples of Hungarian capsicum yielded the following results: *Pericarp*: water, 16·6; ether extract (on the dry substance), 14·6 to 15·0 per cent.; iodine value of the oil, 134. *Seeds*: water, 6·0; ether extract, 31·8 to 32·4 per cent.; iodine value of the oil, 140. *Green Portions* (stalks, etc.): water, 9·1; ether extract, 2·5 to 2·6 per cent.; iodine value of the oil, 110·0 to 112·6. Analyses are also given of specimens of Turkish, Spanish, Japanese, and Indian capsicums; the quantities of the various constituents varied considerably, the ether extract ranging from 9·2 to 10·47 per cent., and the ash from 8·19 to 9·46 per cent. for the fruit free from seeds, whilst samples of ground capsicums yielded from 15·5 to 22·08 per cent. of ether extract and from 5·63 to 9·86 per cent. of ash. As regards the detection of added oil in ground capsicums, the author is of opinion that this cannot be done by taking the iodine value of the oil extracted from a sample. As this added oil would remain on the outer portions of the particles constituting the powder, whilst the natural oil would be still partly enclosed in the cellular tissue, it might be possible to separate the added oil by shaking with water, and the authors are carrying out experiments in this direction. It is pointed out that the United States regulations require that ground capsicums, excluding seeds, shall yield not less than 15 per cent. of ether extract; this portion of the fruit, however, according to the authors' analyses, contains only 14·5 per cent. of ether-soluble substances. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Antiseptic Effect of Creosote Oil and Other Oils used for Preserving Timber. J. M. Weiss. (*J. Soc. Chem. Ind.*, 1911, **30**, 1348-1353.)—The author has investigated the behaviour of pure cultures of *B. subtilis*, *Sacch. glutinis*, and *Penicillium sp.*, all of which can grow on moist sterile wood towards three classes of antiseptics: (1) Oils prepared in the laboratory, mostly separated creosote oil with varying proportions of filtered tar or tar acids added; (2) pure substances such as phenol, cresol, naphthalene, anthracene, quinoline, and paraffin; and (3) commercial creosoting oils. In no case was the antiseptic used in higher proportion than 10 per cent. in the medium, the lowest dilution being 0·05 per cent. A culture medium composed of Liebig's extract, peptone, sodium chloride, maltose, lactose, dextrose, and calcium nitrate in agar was used. The author's conclusions are as follows: (1) The neutral oils of creosote are strong antiseptics; (2) the middle fraction of these neutral oils, boiling from 235° to 270° C., is the strongest: the higher boiling oils are considerably weaker, the lower oils slightly so; (3) coal-tar bases having high boiling-points are strong antiseptics; (4) coal-tar acids are very strong antiseptics, rising in efficiency with the boiling-point; (5) the solid hydrocarbons, naphthalene and anthracene, have low antiseptic values; (6) the addition of filtered tar in moderate amounts to creosote does not materially reduce its antiseptic value; (7) paraffin has no antiseptic value; (8) producer gas-tar distillates have lower antiseptic value and are decidedly inferior to the neutral coal-tar oils; (9) coal-tar creosote is greatly superior as an antiseptic preservative to producer gas-tar distillates and petroleum residues. H. F. E. H.

Use of Charcoal as a Clarifying Agent in the Estimation of Sugar in Urine. A. C. Andersen. (*Biochem. Zeitsch.*, 1911, **37**, 262-265.)—The results of a number of experiments carried out by the author show that the addition of hydrochloric acid, as recommended by Bang and Bohmannsson (ANALYST, 1910, **35**, 70), does not prevent the absorption of sugar by blood-charcoal when the latter is employed for clarifying and decolorising urine. When bone-charcoal is used but little absorption takes place, the quantities of sugar found being practically identical with those obtained by using lead acetate or mercuric nitrate as clarifying agents; bone-charcoal, however, decreases the reducing power of normal urine to a less extent than does blood-charcoal. More satisfactory results are obtained if 50 per cent. acetic acid be employed in place of hydrochloric acid, as the absorption of the sugar by either bone or blood charcoal is almost entirely prevented. Forty c.c. of the urine should be treated with 10 c.c. of 50 per cent. acetic acid and 4 grms. of powdered blood-charcoal, shaken for ten minutes, and then filtered. W. P. S.

Observations on the Assay of Digestive Enzymes. H. T. Graber. (*J. Ind. Eng. Chem.*, 1911, **3**, 919-921.)—According to the U.S. Pharmacopœia, the activity of pepsin preparations is to be tested on "fresh eggs," but the author has found that the digestibility of egg albumin varies considerably according to the age of the egg. The albumin of an egg twenty-four hours old is not in a readily digestible condition, and the maximum condition of digestibility by pepsin is only reached when the egg is five to seven days old; after this the digestibility decreases, and about the tenth day it becomes the same as it was on the second day. It is advisable to retain a sample of pepsin standardised against eggs of known ages, and to use the same for control purposes in future work. In digestion experiments with pepsin extremely small variations in acidity cause large differences in the results. In testing the diastatic activity of pancreatin, various kinds of starch show very different results—for instance, a sample of pancreatin may show an activity towards potato starch one-third greater than towards maize starch. In testing papaïn, finely-minced raw beef-steak should be used; the enzyme does not dissolve cooked beef. Ten grms. of minced beef, 0·325 grm. of papaïn, and 85 c.c. of 0·3 per cent. hydrochloric acid, are digested at 52° C. for six hours; the undigested residue, after settling in a measuring-tube for three-quarters of an hour, should not amount to more than 2 c.c. The coagulating power of rennet is influenced by the condition of the milk and its chemical composition, particularly the proportion of calcium salts; also the manner of mixing the rennet with the milk, the kind of cows from which the milk was taken, and, lastly, the temperature at which the milk was kept before and during the test. The milk should not be stirred, but the rennet should be mixed by pouring from one vessel to another. J. F. B.

Diastatic Power of Malt. C. G. Matthews and F. E. Lott. (*J. Inst. Brew.*, 1911, **17**, 638-642.)—The Lintner process for estimating diastase measures only the saccharifying power, since soluble starch is employed, and this the authors consider a defect, as failing to take account of the starch-liquefying power of the extract. Soluble starch, if over-converted, yields results which are too high; while soluble starches prepared from malt, barley, and potato starches all yielded the same result with the pale malt used. The use of potato starch paste in place of soluble starch is shown to be satisfactory for the majority of malts examined, the average difference being 3·5 units Lintner, the maximum discrepancy being a fall of 16 per cent. with a vinegar malt (D.P. 99·5). Two malts of normal diastatic activity showed values of 3 and 5 units higher when starch paste was employed.

The authors describe the method they recommend, which consists in adding 0·75 grm. of finely-ground malt to 500 c.c. of 2 per cent. starch paste, standing one hour at 70° F., checking the action with soda, diluting to 1 litre, and then titrating the maltose formed with Fehling's solution, the grains being allowed to settle out without filtering. For highly diastatic malts the concentration of starch may be increased to 3 per cent. The vinegar malt mentioned above shows a value of 20 per cent. low by this method (2 per cent. starch), and 11 per cent. low with 3 per cent. starch. The other samples examined show an average difference of 7 units Lintner, the new process values being in all cases but one lower than the standard values.

H. F. E. H.

ORGANIC ANALYSIS.

Estimation of Asphalt in Mineral Oils, Pitch, etc. F. Schwarz. (*Chem. Zeit.*, 1911, **35**, 1417-1419.)—The author recommends the use of butanone (methylethyl ketone) for the separation of asphalt from mineral oil distillates, lubricating cylinder oils, etc. Dry butanone has a sp. gr. of 0·805 at 20° C., but its solvent action is too powerful to permit of a proper separation of the asphalt, especially in presence of a large proportion of oil. A litre of butanone dissolves about 110 c.c. of water at the ordinary temperature, and the saturated mixture has a sp. gr. of 0·835 at 20° C. For the treatment of thick mineral lubricants, a preliminary extraction with saturated aqueous butanone, followed by extraction with aqueous butanone of sp. gr. 0·812, affords a satisfactory separation of the hard asphalt. Two to 4 grms. of the oil to be tested are placed in a 200 to 300 Erlenmeyer flask, fitted with a fractionating column tube, together with 40 to 80 c.c. of saturated aqueous butanone; the contents of the flask are boiled with vigorous agitation for one minute, and the solution is decanted off hot through a No. 589 filter, care being taken to leave all the residual oil in the flask. The operation is repeated twice again with 20 to 40 c.c. of the saturated butanone. Next, the oily residue is treated in exactly the same manner, three times, with aqueous butanone of 0·812 sp. gr., the same filter being used and the extracts collected separately or together with those of the first series. Any oily particles on the filter should be thoroughly washed with the final hot extraction liquid. The residue in the flask is then dissolved in benzene or chloroform, and the solution is poured through the same filter into a dish. The residue after evaporation often contains traces of oily matters, which should be removed by treatment with 5 to 10 c.c. of light petroleum spirit (b.p. 30° to 50° C.); the asphalt is filtered off, washed with the same spirit, dissolved in chloroform or benzene, and collected in a tared dish; it should be quite hard and brittle at 100° C. The results are usually lower than those obtained by the alcohol-ether method, but the latter generally yields soft asphalts containing bodies not included in the hard asphalt separated by the butanone method. The butanone method may likewise be employed for the fractionation of solid bituminous materials and pitch residues. About 1 grm. of the bitumen, soluble in chloroform, is treated as described four or six times, each time with 20 c.c. of butanone of sp. gr. 0·812. The extraction with the saturated aqueous butanone of sp. gr. 0·835 may be omitted with these materials, since the quantity of oily constituents is relatively small. The extracts are filtered hot, and the filter is washed with the same solvent; the asphalt is then dissolved in chloroform and filtered into a tared dish. The matters in the butanone extract may be further fractionated by saturating the extract with water (about 10 c.c. of water per 100 c.c.). The precipitated resins are filtered off, washed with saturated aqueous butanone, and weighed separately, the oily constituents being estimated by evaporating the butanone extract. J. F. B.

Analysis of Mixtures of Ceresin and Paraffin Wax. N. Chercheffsky. (*Ann. de Chim. anal.*, 1911, **16**, 456-463.)—The lowering of the melting-point caused by the addition of paraffin wax to ceresin is sometimes disguised by adding carnauba wax or a similar product. Hence it is necessary in the first place to separate the unsaponifiable matter in the sample by the usual methods. The following table shows the comparative solubility of a typical paraffin wax (melting-point 52° to 54° C.) and of a pure Galician ozokerite ceresin (melting-point 68·7° C.) in different solvents at 15° C.

Solvents.	Pure Ceresin (m.pt. 68·7° C.).	Paraffin Wax (m.pt. 53·7° C.).
	Grms. per Cent.	Grms. per Cent.
Ethyl alcohol, 96·5 per cent.	0·056	0·108
Methyl alcohol, 99·25 per cent.	0·016	0·004
Amyl alcohol	0·100	0·496
Carbon bisulphide	1·9716	19·072
Carbon tetrachloride	1·948	11·784
Chloroform	1·276	5·332
Crystallisable benzene	0·784	4·540
Acetone	0·028	0·120
Ether	0·592	1·920
Petroleum spirit (sp. gr. 0·710)	1·416	5·940

The critical temperature of solution is best estimated in 96·5 per cent. alcohol, and a table is given showing that the results obtained with mixtures of the two waxes are in close agreement with the calculated values. The turbidity temperatures given by 2 c.c. of the melted sample in 4 c.c. of the solvent in a tube 7 to 8 cm.

in length by 1 cm. in diameter also afford valuable indications, as is shown in the following results in ° C., obtained with the same samples of waxes:

	Amyl Alcohol.	Carbon bisulphide.	Carbon tetrachloride.	Chloroform.	Benzene.	Toluene.	Xylene.	Petroleum Spirit (Sp. gr. 0·6886).	Petroleum Spirit (Sp. gr. 0·8725).
Ceresin...	66·4	40·3	42·1	41·8	47·4	41	46·2	45·1	51·3
Paraffin Wax...	51	14·8	23·5	24·9	24·7	24·5	26·4	28·8	29·8

In most cases benzene (boiling-point 80·5° C.) will be found the most satisfactory solvent for this test, and it may even be used to effect a partial separation of ceresin from paraffin wax. The general physical characters of certain typical paraffin waxes and ceresin are summarised in the following table:

Wax.	Origin.	Melting-point.	Refractive Index—		Critical Temperature of Solution in Alcohol (96·5 per Cent.).	Turbidity Temperature in Benzene.	Solubility in Carbon bisulphide at 15° C.
			At 78° C.	At 100° C.			
		° C.			° C.	° C.	Grms. per Cent.
Paraffin 3 RB	America	53·7	1·4280	1·4185	148	24·7	19·07
,, from Schist 3 R	Autun	42·1	1·4238	1·4152	136·5	—	38·68
,, ,, ,, 4 R	Autun	46·5	1·4246	1·4161	141	—	34·65
Ceresin	Galicia	68·7	1·4352	1·4268	172·5	47·4	1·97

C. A. M.

Chemical Examination of Calabar Beans. A. H. Salway. (*J. Chem. Soc.*, 1911, 99, 2148-2159.)—The ripe seed of *Physostigma venenosum* contains, in addition to the alkaloid physostigmine or eserine, some new substances which the author describes. He obtained two interconvertible modifications of physostigmine itself, $C_{15}H_{21}O_2N_3$, which appears to be dimorphous, one melting at 86° to 87° C., and the other at 105° to 106° C. The total amount separated amounted to 0·179 per cent. of the bean. Physovenine, $C_{14}H_{18}O_3N_2$, a new alkaloid, melts at 123° C., and has, like the previous substance, a powerful myotic effect on the pupil of the eye. Another alkaloid eseramine melts at 245° C. Calaberol, $C_{23}H_{36}O_4$, a new dihydric alcohol, melts at 245° C. Trifolianol, $C_{21}H_{36}O_4$, is a dihydric alcohol previously isolated from red clover flowers. Stigmasterol, $C_{30}H_{48}O$, and sitosterol, $C_{27}H_{46}O$, were obtained, and in addition glycerides of behenic, stearic, palmitic, oleic, and linolic acids, and a sugar yielding *d*-phenylglucosazone (melting-point 205° C.). H. F. E. H.

Aromatic Grass Oils. Part I. (*Bull. Imp. Inst.*, 1911, **9**, 240-253.)—These volatile oils are derived from members of the genera *Cymbopogon*, *Andropogon*, and *Vetiveria* (Nat. Ord. *Graminaceæ*), and the chief are citronella, lemon grass, palmarosa, and vetiver oils. The "total alcohols" were calculated from the acetylated oil, and the amount of geraniol recorded was found by the phthallic anhydride method, the citronellal being the difference between these two amounts. Three kinds of grass yield oils of the citronella type: (1) The wild mana grass; (2) the cultivated maha-pengiri grass; and (3) lena-batu grass:

	Mana Grass Oils.							Lena-Batu, *Cymbopogon nardus* (Rendle, Lena-Batu).	Maha-Pengiri, *Cymbopogon winterianus* (Jowitt).
	Oils from *Cymbopogon nardus*, var. *Linnæi* (*typicus*).				Oils from *Cymbopogon nardus*, var. *convertiflorus*.				
	Maha-naran-pengiri Oil.	Heen-naran-pengiri Oil.	"Small-Leaved Mana" Grass Oil.	"Very Broad-Leaved Mana" Grass Oil.	"Glaucous-Leaved-Mana" Grass Oil.	"White-Stemmed Mana" Grass Oil.	"Red-Stemmed Mana" Grass Oil.		
Yield of oil, per cent.	0·180	0·200	0·220	0·170	0·450	0·240	0·160	0·420	0·500
Sp. gr. $\frac{15^\circ \text{ C.}}{15^\circ \text{ C.}}$ ...	0·920	0·913	0·906	0·926	0·913	0·908	0·929	0·913	0·890
Optical rotation in 100 mm. tube at 21° C.	- 3° 7′	+2° 35′	+3° 7′	+1° 31′	+12° 12′	+1° 27′	+6° 19′	- 12° 26′	-2° 45′
Total alcohols, per cent.	51·6	43·5	57·0	48·7	46·5	54·8	39·1	57·8	79·0
Geraniol, per cent.	27·6	24·6	34·4	25·8	29·3	30·2	19·4	31·5	32·5
Citronellal, per cent.	24·0	18·9	22·6	22·9	17·2	24·6	19·	26·3	46·5
Volume of 80 per cent. alcohol producing clear solution	1	1·3	1	1	1	1	1	1	1·2
Volumes of 80 per cent. alcohol required to produce turbidity or opalescence	10	10	10	10	10	10	10	10	Remains clear with ten volumes

H. F. E. H.

New Philippine Essential Oils. B. T. Brooks. (*Philippine J. Science*, 1911, **6**, 333-351.)—*Oil of Michelia Champaca* (*L*) and of *Michelia Longifolia* (*Bl*) (Analyst, 1912, 28).

Oil of Toddalia Asiatica (*L.*) *Kurz* (*T. Aculeata Pers.*).—The leaves of this species yield 0·08 per cent. of oil, of sp. gr. 0·9059 at 30° C., and $[n]_{D\ 30^\circ\text{ C.}} = 1{\cdot}4620$. It consists chiefly of linalol, together with about 18 per cent. of a volatile crystalline compound of camphoraceous odour melting at 97° C.

Oil of Clausena Anisum-Olens (*Bl.*) *Merr.*—The oil from the fresh leaves (1·2 per cent.) has the following constants: Sp. gr. at 30° C., 0·963; $[n]_{D\ 30^\circ\text{ C.}} = 1{\cdot}5235$;

saponification number, 3·6. No optical activity. Ninety-one per cent. distils between 212° to 214° C., and consists of methyl-chavicol. *Clausena* and rosemary are used in the preparation of aromatic liquors.

The oil from Limnophila species (*Scrophulariaceæ*) has a sp. gr. of 0·850, indicating a high proportion of terpenes.

Citrus Oils.—Philippine orange-peel oil is of the usual character: $[n]_{\mathrm{D}\ 30^{\circ}\mathrm{C.}}=1{\cdot}4700$; $[\alpha]_{\mathrm{D}\ 30^{\circ}\mathrm{C.}}$, 90°·85; ester number, 8·0; limonene, about 92 per cent. The freshly-pressed oil deposits about 0·3 per cent. of a crystalline stearoptene.

Citrus Decumana.—The oil of the leaves of this species, which yields the well-known grape fruit, is present to the extent of 1·7 per cent., and has sp. gr. at 30° C., 0·870; ester number, 10; $[\alpha]_{\mathrm{D}\ 30^{\circ}\mathrm{C.}}$, 22°·9; $[n]_{\mathrm{D}\ 30^{\circ}\mathrm{C.}}=1{\cdot}4644$. It is miscible with 70 per cent. alcohol. The oil contains 25 per cent. dipentene, 15 per cent. linalol, and aldehydes in small quantity (? citral). The oil from *C. hystrix* (0·08 per cent.) has sp. gr. at 30° C., 0·915; $[n]_{\mathrm{D}\ 30^{\circ}\mathrm{C.}}=1{\cdot}4650$; $[\alpha]_{\mathrm{D}\ 30^{\circ}\mathrm{C.}}=-10^{\circ}{\cdot}5$; saponification number, 50·2. A. R. T.

Reaction for Detecting Sesamé Oil in Other Oils. Utz. (*Chem. Rev. Fett- u. Harz-Ind.*, 1911, **18**, 292-293.)—Guarneri's test for sesamé oil (*Chem. Zentralbl.*, 1909, II., 869) consists in treating the sample with a few drops of an ethereal solution of hydrogen peroxide and a little nitric acid of sp. gr. 1·4, when a blue coloration should be obtained in the presence of sesamé oil. The author describes a series of systematic tests made upon different oils and fats, from which he concludes that the colours obtained in the case of mixtures are not sufficiently distinctive, and that the reaction is not sensitive enough. The Baudouin test and Soltsien's tin chloride reaction are much more reliable and sensitive than Guarneri's reaction.

C. A. M.

Estimation of Phenol and *p*-Cresol in the Presence of Each Other. H. Ditz and F. Bardach. (*Biochem. Zeitsch.*, 1911, **37**, 272-312.)—Results of a series of investigations are given with the object of confirming the accuracy of the method proposed some years since by Ditz and Cedivoda (ANALYST, 1900, **25**, 74). The method is reliable, provided that the prescribed procedure be followed, and is stated to be more trustworthy than that described by Siegfried and Zimmermann (ANALYST, 1911, **36**, 79). W. P. S.

New Method for Determining the Melting-Point of Pitch. H. F. French. (*J. Ind. Eng. Chem.*, 1911, **3**, 907-910.)—The method consists in heating a suspension of the pulverised material in a liquid until it coagulates. The liquid is heated by the passage of an electric current in an Erlenmeyer flask, which is rotated during the period of observation. The electrodes are $\frac{3}{16}$ inch copper rods encased in rubber tubes and tipped with lead at the bottom; these are bound together with a thermometer. A 220-volt direct current circuit is used, and the electrolyte is dilute sulphuric acid standardised so that a current of 2·2 ampères passes at 25° C., whilst the flask is rotated. The correct grinding of the material is of extreme importance: 20 grms. of pitch are ground until the powder passes through a 40-mesh sieve of No. 32 wire, having holes 0·0137 inch in size. Two grms. of the freshly-ground pitch are placed in the flask with 50 c.c. of the electrolyte and thoroughly shaken; the electrodes and thermometer are introduced and fixed parallel to one of the sides of the flask, which is then rotated at a high speed whilst the current is passed. When the suspended pitch coagulates to a distinct clot the current is switched off and the temperature noted. It is important that the pitch should not be kept longer than an hour or so after grinding, as the melting-point rises considerably on keeping. J. F. B.

Soot: Its Character and Composition. J. B. Cohen and A. G. Ruston. *J. Soc. Chem. Ind.*, 1911, **30**, 1360-1364.)—The character of coal, method of combustion, and distance from place of combustion, all influence the composition of soot. The table shows the main differences observed in domestic and boiler samples of soot:

	Ash, per Cent.	Carbon, per Cent.	Tar, per Cent.	Nitrogen, per Cent.	Number of Lbs. per Bushel.	Value per Ton calculated from Nitrogen.			Acidity, per Cent.
						£	s.	d.	
Boiler, base	75·0	16·7	0·09	—	35·8		—		1·33
,, top	62·0	27·0	1·66	1·21	34·3	0	14	6	0·56
Brass foundry, liquid fuel, base	82·0	11·4	1·02	0·11	63 3	0	1	2	—
Brass foundry, liquid fuel, top	31·4	47·7	10·62	2·32	40·1	1	7	0	0·65
Domestic, kitchen ...	17·8	52·3	12·5	4·11	22·0	2	9	4	0·28
,, room 5 feet up	5·1	36·5	34·9	6·89	16·5	4	2	8	0·92

The lighter and more bulky the soot, the higher, as a rule, is the nitrogen content, and it is advisable to buy by the bushel rather than by weight. The purchaser should demand that there be at least 4 bushels to the hundredweight. The average loss of coal in the form of soot is 6 per cent. in the case of domestic consumption and 0·5 per cent. for factories. The average deposit of soot over the City of Leeds is 220 tons per square mile per annum, the amount of tar varying from 4 to 110 hundredweight per square mile per annum according to locality. The adverse effect of soot in the air on the growth and development of plants is very marked, the assimilating power of leaves having been found to fall from a standard of 100, two and a half miles from the city, to 42 and even 12 in the University and the city itself respectively. The weight of lettuce crops was reduced from 140 to 44 over the same area. The acidity of soot is such that the corrosion of rails is six times as great in towns as in the country, this acidity having a very injurious effect on soil bacteria, and therefore on the fertility of town soils, when present in excessive amounts. H. F. E. H.

New Process for the Estimation of Sugars. F. von Fillinger. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 605-607.)—For the estimation of reducing sugars in such substance as blood, etc., the author describes a process which is a combination of Bang's and Pavy's methods. The following solutions are required: (1) Potassium thiocyanate, 250 grms.; potassium carbonate, 250 grms.; and potassium hydrogen carbonate, 25 grms., per litre. (2) Crystallised copper sulphate, 4·278 grms. per litre. (3) Potassium thiocyanate, 200 grms.; potassium carbonate, 250 grms.; potassium hydrogen carbonate, 50 grms.; and crystallised copper sulphate, 10·42 grms., per litre. The sugar solution under examination should contain approximately 0·1 per cent. of reducing sugars; in the case of solutions of unknown concentration, a preliminary estimation is made by boiling small definite amounts for two minutes with quantities of 10 c.c. of solution (3). The amount of sugar solution which just decolorises the 10 c.c. of reagent solution contains 0·01 grm. of sugar, and the strength of the solution is adjusted accordingly to make it contain 0·1 per cent. of sugar. For the actual estimation 20 c.c. each of solutions (1) and (2) are placed in a flask, and the flask is closed with a rubber stopper, through which passes the nozzle of a burette containing the sugar solution. A side-tube on the neck of the flask is connected by means of a T-piece with two small wash-bottles containing alkaline pyrogallol solution; the tubes in these wash-bottles are so arranged that the air leaving the flask must pass through one of the bottles, whilst air entering the flask passes through the other. The contents of the flask are now boiled until all air has been expelled from the flask, and the sugar solution is then run in gradually until the blue colour has been discharged, the contents of the flask being kept at a boiling temperature meanwhile. The percentage quantity of sugar is found by dividing 1 by the number of c.c. of sugar solution used. For the estimation of sugar in blood or other fluids containing proteins it is recommended that the latter be removed by treatment with ferric hydroxide; from 3 to 4 grms. of sodium chloride may also be added to the 40 c.c. of mixed reagent solutions in order to render the results more concordant.

W. P. S.

INORGANIC ANALYSIS.

Estimation of Antimony in Red Rubber Goods. W. Schmitz. (*Gummi-Zeit.*, 1911, **25**, 1928-1930; through *Chem. Zentralbl.*, 1911, II., 1710.)—Three grms. of the finely rasped rubber are treated in a Kjeldahl flask with 40 to 45 c.c. of strong sulphuric acid. A small quantity of mercury or mercury salt is added, and a piece of paraffin wax about the size of a pea. The mixture is heated until the rubber is dissolved and the black liquid begins to clear; 2 to 4 grms. of potassium sulphate are then added, and the heating is continued until a colourless liquid is obtained. After cooling, 1 to 2 grms. of potassium metabisulphite are added, and an excess of tartaric acid; the liquid is diluted sufficiently to prevent the charring of the tartaric acid, and boiled until the odour of sulphur dioxide has disappeared. A few c.c. of dilute hydrochloric acid are added, the liquid is diluted to 200 c.c., filtered through a dry filter, and 195 c.c. are titrated either with iodine and thiosulphate or, more simply, with potassium bromate, the latter titration being made in acid solution. The above procedure gives accurate results, and is far simpler than the usual gravimetric method for the estimation of antimony. Decomposition of the rubber by means of fuming nitric acid in presence of sulphuric acid gives good results, but the method recommended by Frank and Birkner (ANALYST, 1910, **35**, 131), in which ammonium persulphate and nitric acid are employed, is difficult in operation.

J. F. B.

Estimation of Antimony in Red Rubber Goods. W. Schmitz. (*Gummi-Zeit.*, 1911, **25**, 2002-2003; through *Chem. Zentralbl.*, 1911, II., 1711.)—The method recommended by the author (see preceding abstract) is preferable to that of Frank and Birkner (ANALYST, 1910, **35**, 131), because it can be performed on a relatively large quantity of material (2 to 4 grms.) and because in the latter method the rubber tends to ignite unless the nitric acid be suitably diluted. The oxidation of the rubber by sulphuric acid can, however, be accelerated by the use of ammonium persulphate. When the rubber is completely dissolved and the black liquid begins to clear, solid ammonium persulphate is added to the contents of the flask until the liquid becomes colourless. If small particles of carbon remain floating in the clear liquid, heating must be continued and more persulphate added. The liquid is then diluted and boiled for a long time in order to destroy the residual persulphate. When using this method, the titration with iodine is more suitable than that with potassium bromate, since the persulphate sometimes yields a liquid of yellow colour. The author's method possesses the advantage of being extremely rapid (total time no more than two hours), and of avoiding the presence of nitric acid and oxides of nitrogen, which would interfere with the titration. J. F. B.

Detection, Separation, and Estimation, of Arsenic and Antimony. G. Bressanin. (*Boll. Chim. Farm.*, 1911, **50**, 691-694; through *Chem. Zentralbl.*, 1911, II., 1964.)—The method of Blattner and Brasseur (ANALYST, 1904, **29**, 167) for the estimation of small quantities of arsenic in hydrochloric acid and sulphuric acid yields inexact results: 2 per cent. low, for example, in concentrated hydrochloric acid. With sulphuric acid (sp. gr. 1·45), even if it contained one-third hydrochloric acid, accurate results were obtained. Washing the precipitate of arsenious iodide with hydrochloric acid containing potassium iodide involves the danger of liberating iodine; and 2·5 c.c. or less of the potassium iodide solution should be used instead of the 5 c.c. prescribed. To determine the arsenic in hydrochloric acid, the author mixes 1 part of the acid with 2 of sulphuric acid (sp. gr. 1·45). In sulphuric acid free from lead or tin, the precipitation is carried out without the addition of hydrochloric acid. Commercial sulphuric acid is first brought to a sp. gr. of 1·45, and one-third of hydrochloric acid added; nitroso compounds, if present, are destroyed with urea. For washing, a mixture of 4 parts of sulphuric acid (sp. gr. 1·45) and 1 part of hydrochloric acid is used. The sulphuric acid under examination may be over a sp. gr. of 1·45—*e.g.*, 1·52. Thus modified, the method gave good results. Antimonious iodide was found to be soluble in concentrated hydrochloric acid, and the method, for antimony, is thus confined to estimations in sulphuric acid. The reactions may be followed under the microscope: the yellow hexagonal plates of arsenious iodide are easily distinguished from the purple-red antimony iodide crystals of similar shape. To determine arsenic and antimony occurring together, the sample is dissolved in sulphuric acid (sp. gr. 1·52) to a standard volume, the antimony iodide and arsenious iodide precipitated in an aliquot part, without the addition of hydrochloric acid, and the arsenic and antimony determined together by titration. The precipitate from a second portion to which one-third hydrochloric acid had been added is washed three times in the usual manner, dissolved, neutralised, and the arsenious iodide titrated; the antimony iodide is found by difference. Arsenates and antimonates are estimated similarly. O. E. M.

Detection and Estimation of Arsenic in Organic Compounds. G. Bressanin. (*Boll. Chim. Farm.*, 1911, **50**, 727-730; through *Chem. Zentralbl.*, 1911, II., 1695.)—The author applied the method (see preceding abstract) to the estimation of arsenic in sodium cacodylate, arrhenal, atoxyl, and salvarsan. In sulphuric acid of sp. gr. 1·45, these compounds, except salvarsan, even in dilute solution, gave characteristic, various-coloured precipitates with potassium iodide. The cacodylate always gave the smell of cacodyl. For estimation, 0·2 to 0·4 grm. of the substance was heated carefully in a long-necked flask for two hours with 10 c.c. concentrated sulphuric acid. Sulphur dioxide was expelled by a strong current of air, the contents of the flask transferred, with repeated washings with sulphuric acid (1·45 sp. gr.), to a 100 c.c. flask, and made up to the mark with the same acid. The arsenic was determined as described (see preceding abstract) in an aliquot portion. Results for sodium cacodylate, arrhenal, atoxyl, and salvarsan, agreed well with the theory. Salvarsan, unlike other organic arsenic compounds, in solution in sulphuric acid, gives no precipitate with potassium iodide. It reacts violently with sulphuric acid of sp. gr. 1·84 with evolution of sulphur dioxide; if then diluted with sulphuric acid of 1·45 sp. gr., it gives a precipitate with potassium iodide. O. E. M.

Determination of Copper: A Modification of the Iodine Method. C. Kendall. (*J. Amer. Chem. Soc.*, 1911, **33**, 1947.)—The modification is intended to make the iodide method as rapid as the less accurate, but hitherto more convenient, cyanide method. The removal of the nitrous acid formed by solution of the copper, as metal, oxide, or sulphide, is effected by means of sodium hypochlorite, instead of by evaporation to dryness, or by the use of bromine. The chlorine liberated is converted into chlorophenol, which is not ionised. If the sample is metallic copper, 200 to 300 mgrms. are dissolved in 5 to 10 c.c. of 50 per cent. nitric acid; if cuprous oxide, it is collected on asbestos in a Gooch crucible, and thence dissolved with 10 to 15 c.c. of 30 per cent. nitric acid; if cuprous sulphide, it is collected on a $\frac{1}{8}$-inch layer of asbestos in a Gooch crucible, the crucible with its contents boiled in a 50 c.c. beaker with 10 c.c. of 50 per cent. nitric acid until solution is complete, and solution and asbestos washed into a conical flask; if a solution of a copper salt, a quantity containing 200 to 300 mgrms. of copper is taken. To the solution in a 300 c.c. conical flask, its volume being between 50 and 60 c.c., its acidity equal to 4 or 5 c.c. concentrated nitric acid, and its temperature not above 25° C., are added 5 c.c. of hypochlorite solution made by boiling together 112 grms. calcium hypochlorite having 35 per cent. or more available chlorine, 100 grms. anhydrous sodium carbonate, and 1,200 c.c. water, filtering, and adjusting the hypochlorite solution until 5 c.c. are equivalent to 30 c.c. $\frac{N}{10}$ thiosulphate. A change of colour from a clear blue to a greenish tint, or the evolution of chlorine, shows that enough hypochlorite solution has been added. At the end of two minutes, 10 c.c. of colourless 5 per cent. phenol are rapidly added, chlorine above the liquid blown off, the flask washed down with water, and 20 per cent. sodium hydroxide run in until a very slight precipitate forms. This is just dissolved by the addition of 50 per cent. acetic acid, followed by 10 c.c. of potassium iodide solution containing 30 grms. per 100 c.c. The titration is made with thiosulphate (1 c.c. = 6 mgrms. Cu) until the liquid has a light straw colour. After addition of starch solution the titration is finished, thiosulphate 1 c.c. of which is equivalent to 1 mgrm. copper being used if great accuracy is required. The results appended show a percentage error ranging from −0·10 to +0·15. O. E. M.

Method for the Estimation of Selenium in Sulphur and Pyrites. P. Klason. (*Chem. Zeit.*, 1911, **35**, 1345.)—Sulphite lye containing 0·5 mgrm. selenium per litre is useless. The author estimates this small quantity by means of hydriodic acid: the selenious acid sets free iodine, which is detected by its action on starch. As a control, sulphur to which a known quantity of selenium had been added was burnt in a tube. Until all the sulphur was burnt, no oxidation of selenium took place; then, in oxygen, selenium dioxide was sublimed, and it was found that 0·1 mgrm. could be detected with certainty. Pyrites was treated similarly, but in a quartz tube. Sicilian sulphur was found to contain about 0·9 grm.; Japanese, 1 to 20 grms.; and Falun pyrites, usually 10, but sometimes 30 or 40 grms. selenium per ton. The author's method allows 92 per cent. of the selenium in sulphite liquor containing only 0·25 mgrm. selenium per litre to be determined. O. E. M.

Volumetric Estimation of Sulphur in Iron and Steel. T. G. Elliot. (*Chem. News*, 1911, **104**, 298.)—A mixture of 5 grms. of the sample with 0·25 grm. finely powdered anhydrous potassium ferrocyanide is wrapped in one 11 cm. filter-paper if the sample is a graphitic iron, in two if it is a steel or white iron, annealed in a covered porcelain crucible for twenty minutes in a closed muffle at 750° to 850° C., and allowed to cool slowly outside the muffle. If the paper is burnt away from above the drillings, the annealing has been at too high a temperature or too long, and the results will be low. The drillings are emptied into a glass mortar, loosened with a pestle, and transferred to the evolution flask, without touching its neck or sides. With the flask is connected a condensing-tube, 6 inches by 1 inch, containing 2 inches of water and standing in cold water, and with this in turn a flask containing 60 c.c. of cadmium chloride solution, made by dissolving 20 grms. of cadmium chloride in water containing a few drops of hydrochloric acid, adding ammonia until the precipitate formed dissolves, acetic acid to acidity and 20 c.c. in excess, and water to 2 litres. A safety-flask containing more cadmium solution is attached. To the sample are now added 50 c.c. of concentrated hydrochloric acid, and heat is applied until the frothing shows that solution is well begun. The flame is lowered until the stream of gas slackens, then raised, and the liquid gently boiled until appreciable evolution of gas ceases. Excess of standard iodine solution is run into the absorption-flask—usually nothing is found in the safety-flask—then about 10 c.c. of 1 in 2 hydrochloric acid, and the sulphide dissolved by shaking. The excess of iodine is titrated back with standard sodium thiosulphate. Displacement of the air by CO_2 is unnecessary in works practice. Correction may be made for any sulphur introduced as sulphates in the paper or ferrocyanide from the difference in sulphur found in samples of the same pure iron with and without annealing. The standard solutions used (1 c.c. = 0·005 per cent. sulphur on a 5-grm. sample) are standardised against a steel or iron of sulphur-content determined by the "aqua regia" or Bamber's method. An iron containing 0·2 per cent. of titanium and 0·080 per cent. sulphur (gravimetric) gave 0·018 unannealed, 0·066 after annealing at 800° C., and 0·076 per cent. after annealing at 950° C. for five minutes and above 790° C. for fifteen minutes. This suggests that with proper preliminary treatment the evolution process may be applied even to titanium pigs. O. E. M.

APPARATUS, ETC.

The Quantitative Manipulation of Small Quantities of Precipitates.

J. Donau. (*Monatsh. für Chem.*, 1911, **32**, 1115-1139.)—The author describes the apparatus and methods adopted in carrying out his improved method of quantitative microchemical analysis, and gives details of procedure in the case of twenty-six typical gravimetric estimations. The Nernst microbalance has been improved by constructing the beam and pointer in a single piece, with a glass counterpoise fused on to the beam at the end near the pointer. At the other end of the beam a piece of silver wire is fused into the glass, from which the filter-pan is suspended by means of a strand of very fine Wollaston wire, ending in a hook below. The whole balance is enclosed in an air-tight case sealed with paraffin wax to exclude moisture; the atmosphere inside is kept dry by means of quicklime, and access is obtained to the pan-hook by a circular hole 3 cm. in diameter, closed by a glass slide which is smeared with vaseline. For collecting the precipitates, the asbestos filter-bed (Analyst, 1911, **36**, 180) has been replaced to great advantage by one of spongy platinum. The filter-pan is formed out of a circular piece of thin platinum-foil 10 to 12 mm. in diameter, perforated at the bottom and provided with a wire loop for hanging on the balance. Spongy platinum is deposited on the perforated bottom of the pan, and the deposit is covered with a smaller platinum-foil tray, also perforated; in this way most of the precipitate, after weighing, may be lifted out of the filter bodily, leaving only a little to be dissolved out by chemical means.

The author describes an ingenious arrangement for producing and manipulating the precipitates. It consists of a short glass tube 1 cm. wide, open at the top, and terminating in a narrow tube at the bottom, this being closed by a piece of glass rod attached by a rubber tube. The substance is dissolved in this vessel, the precipitating reagent is added, the whole being heated if necessary. The upper mouth of the precipitating vessel is then closed with a cork carrying a glass bulb-tube which has just been heated. On cautiously opening the lower end, air is sucked up the capillary, and the bubbles stir the precipitate. Similarly, by applying a hot glass od to this bulb-tube the liquid and precipitate can be expelled down the capillary into the filter. A bell-jar is placed between the pump and filter. The precipitating vessel is washed out by similar procedure described above. Full details are given in the paper. J. F. B.

Apparatus for Extracting Liquids with Immiscible Solvents, and for Measurement of Evolved Gases. R. F. Bacon and P. B. Dunbar.

(*U.S. Bureau of Chemistry Circular*; through *Chem. Eng.*, 1911, **14**, 430-431.)—*Continuous Liquid Extraction.*—As shown in Fig. 1, the apparatus consists of a flask, *a*, of glass tubing, of 2 inches in diameter ($\frac{1}{16}$ inch thickness) and 20·5 inches in length, and enlarged to about 3 inches at its lower end; an extraction thimble, *b*, an ordinary test-tube of $1\frac{1}{2}$ inches diameter and of 100 c.c. capacity when filled to $1\frac{1}{2}$ inches of the top, having at a distance of a $\frac{1}{4}$ inch from the top and on opposite sides two holes about $\frac{1}{4}$ inch diameter; a Gooch crucible funnel, *c*, 8·5 inches long, but longer if dense liquids are to be extracted, with the lower end of the stem ground at an angle of 45 degrees; and a condenser, *d*, hanging loosely in the flask. About 100 to 150 c.c. of ether are poured into the flask, and 100 c.c. of the liquid to be extracted is placed in the test-tube; the funnel is inserted into this tube and suspended in the flask, about 3 inches above the bottom, with a copper wire passing through the holes in the tube and hooked over the rim of the flask neck. The condenser is placed in the top of the flask, and condensed ether drops from the condenser into the funnel, and is carried down to the bottom of the tube, whence it passes through the liquid, and overflows at the top. The efficiency of the extractor may be increased by using a glass spiral attached to the stem of the funnel (Kampf, *Chem. Zeit.*, 1910, **34**, 1365).

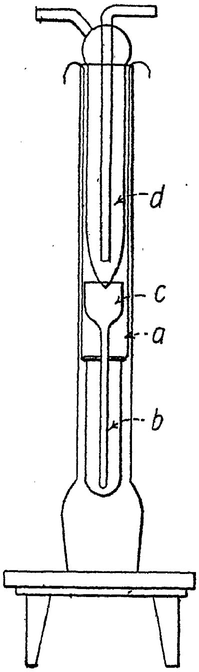

Fig. 1.

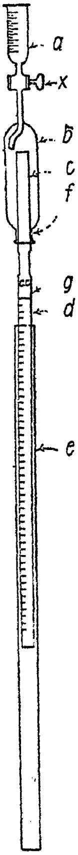

Fig. 2.

Measurement of Evolved Gases.—The solid or liquid substance to be acted on is placed in the reaction chamber, *b* (Fig. 2), and with the stopcock, *x*, open, the liquid in the eudiometer-tube, *d*, is brought to zero by raising or lowering the levelling-tube, *e*. The reagent is placed in *a*, with the stopcock closed, and, after lowering the levelling-tube, the stopcock is opened and a known quantity of reagent mixed with the material to be acted on. The reaction-chamber, *b*, may be heated if required by means of a coil wrapped round it. After cooling to room temperature, the increased volume of the air and gas in the apparatus is read off as usual, deducting the volume of the added reagent. The absorption-tube, *c*, is filled with beads, and is sealed on to the endiometer, and fits into the reaction-chamber, *b*, by means of a ground joint at *f*. The apparatus is suitable for the estimation of citric acid by Spica's method, and for amino-acids by the method of Van Slyke. A. R. T.

Notes on Thermostats, Shaking and Stirring Apparatus, etc. A. C. Cumming. (*Chem. News*, 1911, **104**, 307-309.)—A thermostat is best made of copper, and should be of such size that the introduction of the experimental apparatus does not materially alter the temperature of the contained water. A constant level is maintained in the usual way. The liquid contained in the regulator attached to the thermostat should have a low specific heat, high co-efficient of expansion, and low density. Mercury has a very low specific heat (0·03), but its great density necessitates the use of thick-walled apparatus. Benzene, toluene, alcohol, ether, carbon tetrachloride, etc., may be used, the one having the lowest density being the most efficient. A spiral to give a large surface of exposed liquid is commonly employed, thus giving a sensitive regulator (Lowry, *J. Chem. Soc.*, 1905, **87**, 1030), and this spiral is preferably made of metal. A satisfactory connection is made with rubber if a water-trap is introduced between the mercury and the organic liquid, so that the water layer covers the junction between the metal and glass. Of the forms of apparatus for cutting off the gas-supply, those of W. H. Green (*Chem. News*, 1908, **98**, 49) and T. S. Patterson (ANALYST, 1902, **27**, 234) are

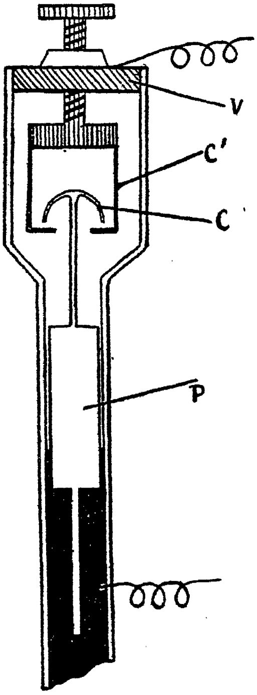

FIG. 1.

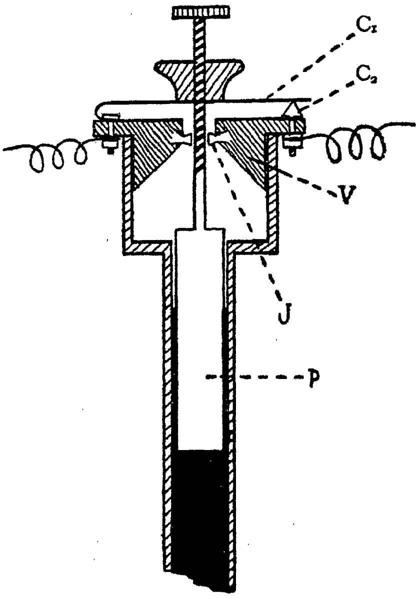

FIG. 2.

satisfactory. A novel method of altering the amount of mercury in the regulator, to produce altered temperature, has been described by A. Slator (ANALYST, 1911, **36**, 127).

Two forms of electrically-controlled regulator have been devised by C. Fraser, and have been found to be reliable. In the first (see Fig. 1), a metal plunger, *P*, floats on the surface of the mercury. This carries a curved platinum wire, *c*, at the end of a rod, and contact is made when *C* comes into contact with *C′*, the point of contact being covered with platinum. The vulcanite plug *V* serves to insulate *C′* and its connections. The adjustment is made as usual, finally by raising or lowering *C′* by means of the brass screw. Fraser regards the form shown in the next figure as an improvement on the latter. Contact between two platinum terminals (C_1 and C_2) is broken when the rise of the plunger lifts C_1. A side-to-side movement is avoided by the garnet jewel setting (*J*) in the vulcanite ring *V*. The regulating screw serves for fine adjustment, the rough alteration being made by increasing or reducing the amount of mercury in the regulator.

In connection with shaking apparatus, J. Knox uses a driving band of brass wire wound in a narrow close spiral. This is advantageous for bands to be used under water, owing to the disintegrating action on leather and gut. For stirring apparatus the author uses the glass-angle stirrer shown in the illustration. The side-tube is 2 inches long, and the vertical tube of such length that it ends about 1 inch from the bottom of the bath when the side-tube is 2 inches below the surface. A deep thermostat may be stirred by blowing a stream of air through the water, the bubbles of air ascending through a wide tube open at both ends. The temperature in any part of the bath is thus kept at not more than 0·1° variation from other parts. More vigorous stirring by mechanical means is, however, necessary for closer constancy of temperature.

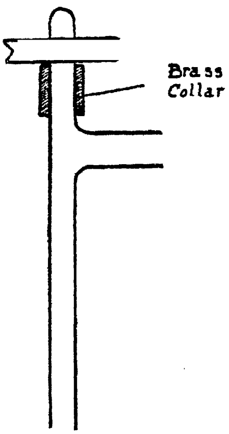

FIG. 3.

A. R. T.

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REPORTS.

On the Work of Inspectors of Foods. A. W. J. Macfadden. (Appendix A, No. 8, from the Annual Report of the Medical Officer of the Local Government Board for 1910-11.)—The Report deals with work in special relation to the Public Health (Regulations as to Food) Act, 1907, meat inspection, food-poisoning, work in special relation to the Sale of Food and Drugs Acts, Conference on Methods of Analysis, Paris, 1910, and with foreign and colonial food legislation. Special inquiries were made in regard to the chemical treatment of flour, and the purity of baking-powders and self-raising flours, and reports dealing with these questions have been issued (*cf.* ANALYST, 1911, **36**, 254, 255). A report was also issued on the occurrence of parasitic nodules in meat exported from Australia (*cf.* ANALYST, 1911, **36**, 129). In order to deal more effectively with this condition in Australia, the Commonwealth Government have taken over the control of the inspection of meat for export, which was carried out previously by the various States. Several cases of illness attributed to the consumption of meat foods containing bacterial poisons occurred during the year; reference is made to the difficulty which is generally met with, of obtaining at a sufficiently early date information of the kind necessary for elucidating the causes of the poisoning.

Representations and reports received by the Board on matters relating to local administration of the Sale of Food and Drugs Acts entailed special inquiries or interviews. Particular notice is directed to the unsuitable procedure adopted in certain districts with regard to the taking of samples. Among other defects may be noted the practice of sampling officers asking for "new milk" when purchasing milk samples for analysis; this simply supplies a gratuitous warning to the vendor to be on his guard. The Sale of Milk Regulations refer to "milk," and the use of the term "new milk" is unnecessary.

Considerable attention has been given by local authorities to the question of lard and lard substitutes. Added water was reported to be present in a few of the samples of lard, and in each of these instances the lard was also found to be adulterated with foreign fat. One sample of "compounded lard," purchased in London, contained 17·8 per cent. of water.

All the samples of imported margarine taken by Customs officers during the year were examined for the presence of paraffin, and one sample imported from Holland was found to contain 3·4 per cent. of solid paraffin. Although the Board have no reason to consider that this form of adulteration is common, they would be glad to receive information as to the results, positive or negative, of any examination for the presence of paraffin which may be made by the public analyst when samples of margarine and lard are submitted to him.

Rules for the unification of the expression of analytical results, formulated by the International Conference, Paris, 1910, are printed in an addendum to the Report (*cf.* ANALYST, 1911, **36**, 536). W. P. S.

The Connecticut Agricultural Experiment Station. (Thirty-third and Thirty-fourth Reports, 1909 and 1910.)—This volume of over 800 pages forms Public Document No. 24 of the State of Connecticut, and is the biennial report for the two years ended October 31, 1910. Almost every department of agricultural activity is embraced, and the sections dealing with plant diseases and entomology are well provided with excellent illustrations and photographs. A very full report on commercial fertilisers, usefully classified, is followed by sections in which a total of 1,630 samples of foods and drugs are summarised, exactly 25 per cent. of these having been found to be adulterated or below stan ard. The methods of analysis employed in the examination of many of the materials dealt with are described. Illegal labelling and misbranding are carefully looked for, as they constitute offences against the State laws. An interesting report is that dealing with the analyses of some fifty named proprietary breakfast foods. It is curious to note that no single examination of coco or chocolate appears in the whole volume, and since the practice of adulterating cocoa with ground shell is known to exist, the omission is unfortunate.

Commercial feeding-stuffs are fully dealt with, useful tables being appended of their composition, digestibility, and price. A very full account of canned peas is a valuable feature in the present work; over 100 samples are described and analysed in detail. The American public, if they choose to avail themselves of reports like the present, are clearly afforded ample information as to the most satisfactory manufacturers to patronise for all the principal materials of domestic consumption, and the practice of publishing names in full should act as a strong inducement to sellers to provide a trustworthy and unadulterated article. H. F. E. H.

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REVIEWS.

A TEXTBOOK OF PRACTICAL CHEMISTRY FOR TECHNICAL INSTITUTES. By A. E. DUNSTAN, D.Sc., Head of the Chemical Department, East Ham Technical College, and F. B. THOLE, B.Sc., Lecturer in Organic Chemistry, East Ham Technical College. Methuen and Co., Ltd. London, 1911. Price 3s. 6d.

The aim of this book is to give a concise account of the methods employed in the analysis and examination of both inorganic and organic compounds in a form suitable for a graded curriculum of instruction in analytical chemistry.

The first portion of the book is devoted to qualitative analysis, the reactions given being all based on the ionic hypothesis, a short and clear account of which precedes the reactions described. The succeeding section on volumetric analysis comprises the methods for preparing the ordinary standard solutions and their simpler applications. For the standardisation of potassium permanganate (p. 147) the use of sodium oxalate might have advantageously been included, as it is more reliable and accurate than either ferrous ammonium sulphate or oxalic acid, which are recommended. The third section of the book deals with the simpler methods of gravimetric analysis and the complete quantitative analysis of some mixtures and compounds. The selection of these is open to some criticism, but a fair choice is available, so that they can be adapted to the capacities of individual students. A reliable method for the separation of iron and manganese, such as the basic acetate method, should have been included, in preference to simply precipitating the iron in a mixture of the two substances, as hydroxide (p. 194), as the latter method can have no claim to accuracy, whilst the former is a really useful analytical method, and a good exercise in analytical procedure.

"Simple Gasometry" is the heading given to the succeeding section, which extends only to ten pages. Whilst one welcomes the inclusion of an outline of the methods employed in this important branch of analytical work in a book of this character, the selection of the apparatus described might have been more suitable. For instance, the use of Hempel pipettes is described in conjunction with the Lunge nitrometer; little additional space would have been required to include a description of the proper burette used for such estimations.

The remaining portions of the book deal with the tests for the elements in organic compounds and their quantitative estimation, some of the commoner organic estimations, familiar operations in organic chemistry—such as crystallisation, distillation, hydrolysis, nitration, etc.—and a chapter on the identification of simple organic substances. The concluding chapter is on physico-chemical determinations, and comprises methods for the determination of molecular weights, conductivity, velocity of reaction, and a description of the use of the polarimeter. The subject-matter included in these sections is carefully selected, and the accounts of the various processes are throughout clear and concise.

The total subject-matter included in the 329 pages of the volume is really remarkable, and its value is enhanced by several very useful tables and by the tabular descriptions of reactions included in the book. The writing is on the whole both careful and accurate, and but few misprints have been noted. Schrötter's carbonic acid apparatus is described as "the Schroedter apparatus" on p. 177, and phenolphthaleïn is written "phenolphthelein" on p. 156. Bearing further in mind the extremely moderate price at which the volume is published, it should prove both helpful and attractive to students in technical schools and institutes, for whom it is more especially written.

CHARLES A. KEANE.

LES MATIÈRES CELLULOSIQUES. By J. G. BELTZER and JULES PERSOZ. Librarie Polytechnique, Ch. Béranger. Paris, 1911. Price 5 francs.

This is a useful handbook, rather technological than strictly scientific, according, it would appear, to the purpose and intention of the authors.

It follows, perhaps too closely, the general plan and basis of classification of the well-known earlier textbooks, but introduces new matter of importance arising out of the investigations of the period 1895-1910. This matter, however, should have been more critically edited, and not reproduced *en bloc*. Thus a section on pp. 281-282, under "Dosage de la Cellulose de bois . . . équivalent d'hydrazine des fibres," describes the method of determining the absorption by fibrous products of phenylhydrazine from solution of the acetate, and of thus fixing a constant of these products, of presumably critical value. In a table of typical results we note the following for phenylhydrazine combining calculated to 100,000 of—

	Extreme Numbers.
Cottons	0·433 to 1·101
Flaxes	6·886 to 10·351
Jute	9·081 to 12·761

The authors introduce the method "pour servir à évaluer le degré de lignification," and the student would draw the conclusion that jute and flax are closely related *quâ* lignification!

There are other lapses which leave the critical reader in doubt as to the cause of confusion.

Thus on p. 292, under "Le Chanvre," Fig. 13, which is a section of a fibro-vascular bundle (monocotyledon), is described as of the hemp-stem, "tige de Chanvre," and representing a kidney-shaped complex of lignified fibres surrounded by parenchyma. The context suggests that only the true hemp (*Cannabis*) is under consideration, and a student would be much misled by this section (pp. 290-294). Of course, it is well known that the term "hemp" is loosely employed in commerce, being also applied to manila, phormium, sisal, etc., somewhat as "linen" is loosely applied, outside Belfast, to cotton and union goods. But the atmosphere of a textbook is supposed to be free from fog.

In the section on flax fibre the authors describe the fibre bundles as so compacted as to require an alkaline boiling for their separation. This description, apparently of only incidental moment, is a misdescription which obscures the entire practice of the flax-spinner.

We do not labour these points; they have to be noted in order to apprise the more critical student that he may be misled even by his teachers.

The second part of the volume, for which M. Persoz is responsible, deals with the analytical investigations of papers. It is in large part a reproduction of the methods adopted, and in part originated, by Herzberg. The author has lost an opportunity of a progressive contribution to the subject. Every practical worker is aware that our laboratory methods are extremely systematic and rapid, and an author, to command attention in his field, should show that his *experience* and knowledge are on the level of advanced practice.

In dealing with sizing agents, the author makes no mention of the chloramine reactions of the proteids. The reaction of the celluloses of the *Gramineæ* with anilin salts is mentioned as a recent discovery of M. Petitgout.

The examination of papers for reactions (acid or basic) is described (p. 423) in terms of the academic method of cutting the paper into strips, boiling with distilled water, and testing the extract with Congo red and litmus. This very important factor of the paper-makers' finish is usually diagnosed by rapid methods, and a gamut of indicators applied to the paper itself.

This section, however, would not appeal to English readers unless it could be commended as evidently progressive, which we are unable to do. On the whole the work is to be regarded as a useful *résumé* of scientific material which is in a condition of active growth. C. F. CROSS.

SOUDE-POTASSE SELS. By P. MÉKER. Collection des Manuels Pratiques d'Analyses Chimiques. Publiès sous la direction de MM. Bordas et Roux. Paris: Ch. Béranger. 1911. Pp. 245. Price 5 francs.

This small analytical textbook, written especially for the use of chemists engaged in the French Customs Department, consists of four separate parts. The first and second portions deal with the analytical methods employed in the analysis of the various sodium and potassium salts occurring in commerce. The methods described are generally of a satisfactory character, but, at the same time, do not present any novelty, and are already available in several English works. The third part is concerned with the analysis of brine, marine salt, and rock salt as imported, and with the analysis of the numerous salts which have been "denatured" in various ways in order to allow of their introduction into France for industrial and agricultural purposes at a lower rate of duty. The fourth section, occupying two-fifths of the book, consists of a reprint of the chief decrees and circulars of the French Customs Department.

Whilst the book will be a useful one to those for whom it is especially written, and also for those interested in the export of such salts to France, it is not one which will be of much use to chemists as a whole. H. G. COLMAN.

THE CHEMICAL WORLD. Edited by W. P. DREAPER. Published by Messrs. J. an A. Churchill. London. Price 6d. monthly.

What is likely to prove a popular addition to the growing literature devoted to chemistry is the *Chemical World*, the first number of which appears this month. As the additional title indicates, the new venture is to be "A monthly journal of chemistry and chemical engineering." To judge by the first issue, it may be fairly admitted that the new journal's claim to "fill a space in the scientific literature of this country which is not at present fully occupied" is justified, no other periodical of the kind covering such a wide field in the application of chemical knowledge. The Editor has succeeded in getting together a representative selection of writers, some twenty or more special articles being contributed by well-known chemists, each of whom is particularly well fitted to deal with his chosen subject. Thus, Dr. Russell presents a *résumé* of the recent work done at Rothamsted and elsewhere on sterilised soils, Dr. Harden treats of Progress in Biochemistry, Mr. G. T. Holloway sums up Metallurgical Progress in 1911, while many others, amongst them being Messrs. C. F. Cross, Revis and Bolton, Jago, and Drs. Kenneth Mees and S. Rideal, all contribute useful short papers upon branches of chemistry which they have made their own. Mr. Emil L. Hatschek's recent lectures at the Cass Institute on "Colloids" are being reprinted, and when completed in the *Chemical World* will be published as one of the *Chemical World* Series of text-books. A feature which should prove of general interest is an illustrated account of the Chemical Department of the Imperial College of Science and Technology, written by Dr. J. P. Philip. This is the first of a series dealing with the Chemical Departments of the Modern Universities. Several books are reviewed, among them being "Cocoa and Chocolate," by R. Whymper. One of the photomicrographic illustrations of this work has been reproduced, entitled "Pure Cocoa," but the only recognisable tissues are some particularly robust spiral vessels which are usually looked upon with suspicion when found in anything claiming to be "pure cocoa." A few misprints are to be found, "mycolderma" for "mycoderma," and "nito" for "nitro," but, speaking generally, the printing and illustrations are good.

H. F. E. HULTON.

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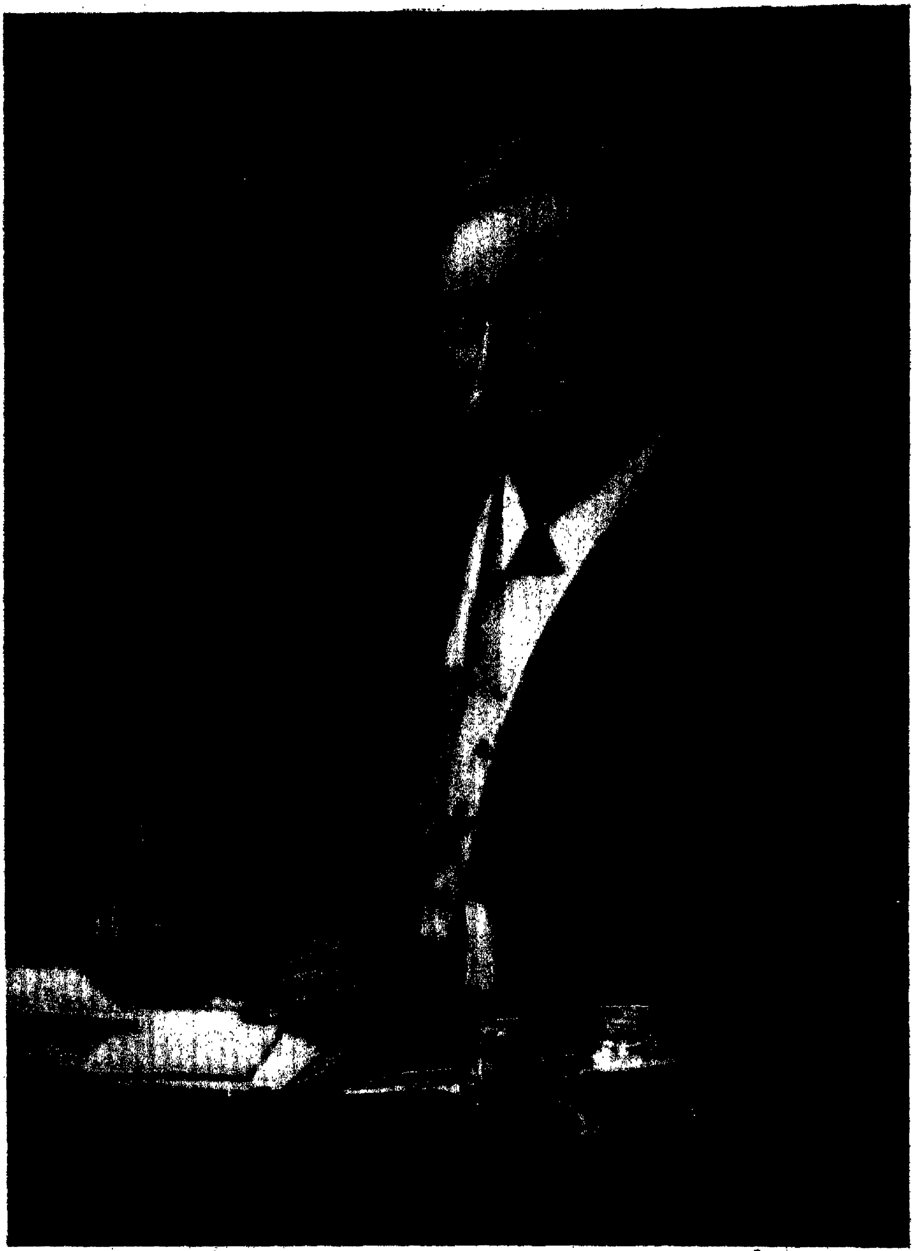
INSTITUTE OF CHEMISTRY.

PASS LIST: JANUARY EXAMINATIONS, 1912.

OF twelve candidates who presented themselves for the Intermediate Examination, seven passed: R. M. Doidge, B.Sc. (Lond.), D. R. Frazer, S. G. Greene, G. S. Heaven, B.Sc. (Lond.), W. Honneyman, A. Wilson, and T. Wright. Of fifteen candidates who presented themselves for the Final Examination, eleven passed. In the branch of Mineral Chemistry: E. C. Evans, B.Sc. (Wales), H. W. Moss, A.R.C.Sc.I., and N. C. Nag, M.A. (Calcutta); in the branch of Organic Chemistry: D. Cardwell, B.Sc. (Manc.), H. Krall, B.A. (T.D.C.), F. H. Lees, and W. M. Roberts, B.Sc. (Manc.); in the branch of the Chemistry of Food and Drugs, and of Water: G. D. Elsdon, B.Sc. (Birm.), H. Hawley, M.Sc. (Birm.), R. L. Morris, and F. G. C. Walker.

ERRATUM.—1912, p. 4, footnote: "16·55 to 17·05" should read "165·5 to 170·5."



H. P. Robinson & Son, Redhill & Guildford, photographers *Emery Walker Ph. sc.*

MARCH, 1912. Vol. XXXVII., No. 432.

THE ANALYST.

OBITUARY.

JOHN MUTER.

All active and energetic men, for whom the working hours are not long enough for the fulfilment of their duties, must feel with Renan that one of the most astonishing things in human life is its shortness. The thoughts span the bridge between youth and age; the road seemed so long, the real distance traversed is so little. It seems but yesterday since the writer of this notice attended, as a young man, in March, 1876, for the first time, a meeting of the Society of Public Analysts in Cannon Street Hotel. The cover of the first number of the Analyst was being shown by the Chairman, Wanklyn, to the members, and a paper was being read by Dr. John Muter on the analysis of butter. This was his first meeting with a man who, then already very well known, was one of the founders of the Society; one of the few who had some practical knowledge of the analysis of drugs and food, and from whom more could be learned than was contained in the then available books on the subjects interesting Public Analysts. Books, indeed, were few and feeble. There was Pereira on "Drugs" and Hassall on "Foods," both dealing mainly in generalities, and breaking down whenever one tried to dive below the surface of any subject. There was an anonymous work, "Death in the Pot," a sensational mixture of fancy with some facts, and a little book by Atcherley, with a collection of analyses of commercial articles, ranging from nuts to nightingales' dung. Knowledge, such as is now amply stored in the tables contained in "Koenig," or on the shelves that groan with the weight of thirty-six volumes of the Analyst and twenty volumes of the *Zeitschrift für Untersuchung der Nahrungsmittel* had not been born.

The little band of men who realised that the co-operation of all practical chemists was necessary in order to carry out the duties imposed upon them by the Sale of Food and Drugs Act that had been passed in 1875, and in the shaping of which they had taken an important share, comprised Dupré, Allen, Wigner, Muter, and Heisch, as the most active of the founders of the Society. The profession of Analytical Chemists was quite small; indeed, it is scarcely correct to speak of such a profession at that time. The lecturers at various hospitals, the professors at colleges and the Pharmaceutical Society, some medical men who had taken, as amateurs, to the microscope and the test-tube stand, formed the bulk of the Analytical Chemists in London. Wanklyn, a few years before, had written in the *Chemical News* epoch-making papers on the composition of milk; these had led to long and bitter controversy, which reverberated for years afterwards. The writer of this notice, with Arthur Angell, had published a pamphlet on a distinction between butter-fat and other fats, which had been promptly ridiculed by some of his experienced elders. At the meeting in Cannon Street in March, 1876, Muter had confirmed its correctness, and hinted that he had long been aware of these facts. It was not easy to grant to a mere boy that he might have ferreted out some knowledge that had escaped the attention of his elders.

In time all these little irritations and jealousies vanished in the common desire for the elucidation of facts, and John Muter became a friend with whom it was a pleasure to consult and co-operate.

John Muter came from a pharmaceutical stock. His father was Professor of Materia Medica in the Portland Street Medical School at Glasgow; his grandfather was the founder of the Glasgow Apothecaries' Company. John was born in Glasgow in 1841. He studied at the Andersonian University, Glasgow, at which the writer, a few years later, in 1873, was co-assistant to one of the lecturers with William Ramsay, who had just come, "promovirt," from Tübingen, and while Dr. Thorpe was professor. Muter became assistant to Dr. Penny. For a time he worked for Dr. Hassall, in which position he was later on succeeded by Alfred H. Allen and the writer, all unknown to each other. In 1872 he founded a laboratory of his own under the title "South London School of Pharmacy," after he had for some years earned a living as a peripatetic science lecturer. His school was mainly established in the first instance for the purpose of coaching pharmaceutical students for the minor examination, then a perfectly mechanical matter. Gradually his teaching developed into thorough and exhaustive courses, comprehensive and up-to-date, and a large number of students obtained from him effectual pharmaceutical training. For their use mainly he wrote a book on analytical chemistry, which ran through a number of editions. Many samples of drugs were sent to him for analysis, and even before the Food Acts had been passed he had founded a considerable chemical practice.

The Society of Public Analysts was formed while food legislation was being discussed before a Parliamentary Committee. In its formation Wigner and Muter bore the main share. The former was an able and most energetic organiser, the latter the more widely experienced practical chemist. Both were rather looked "down upon" by many of the "pure" chemists who then, more or less openly, despised the mere practitioner; indeed, often with good reason. Hence the Society at its start had by no means the good-will of chemists as a whole; difficulties of multitudinous kinds, scientific and otherwise, soon arose; the first President, Dr. Redwood, and several others, resigned; angry discussions and recriminations filled the air; but Wigner and Muter held on.

When the small and penniless Society started their Journal, after the *Chemical News* had hospitably for a year published the proceedings, they found that the names of responsible editors were necessary, to stand the brunt of eventual legal action. In the first year Wigner alone figured on the title-page as editor; from 1877 to Wigner's death, in 1884, Wigner and Muter jointly; and thence till 1891 Muter by himself. The Journal had really been started as the organ of the Society and its property, but, as there was no money, Wigner and Muter financed it and gradually became the *de facto* if not *de jure* owners.

It was far from easy at that time to conduct and fill a monthly journal mainly devoted to food analysis. No other society existed in the world similar to the Society of Public Analysts ; abstracts were not to be had except quite occasionally; original matter had to be created by a few, somehow. Almost every month the energetic secretaries of the Society sent urgent "whips" to a few active members, who, not then overburdened with the duties of their profession, but rather rich in unprofitable time, had ideas and energy and could work up new matter for the meetings. Dupré, Allen, Muter, Wigner, the writer, and one or two others, were the main stays of the young Society and its Journal. Thus, gradually systematic and scientific food analysis was evolved, full of weaknesses and faults of course, but yet infinitely better than anything previously available. Food legislation in other States of Europe and in America followed, and with it the co-operative work of an army of chemists, opening flood-gates of food analytical literature.

Dupré, as the first constitutional President of the Society, was followed in 1879 by Muter, who contributed a considerable number of papers on all sorts of practical subjects, many being of more or less ephemeral interest; others—like his papers on the separation of oleic acid from saturated fat-acids, his method for the estimation of glycerine, on the analysis of condensed milk—of more lasting value. He took an active and instructive share in the discussion of the papers read before the meetings, and his fund of merriment and jollity found full scope at the "informal" adjournments after the meetings at Burlington House, that were invented very early in the history of the Society by the present historian. None of the old members will forget the informal Annual Meetings, not then the serious, staid full-dress functions into which they have inevitably grown—the meetings at which Allen sang his own compositions, Sykes fiddled, John Newlands recited about Garibaldi, and Vieth and the writer duetted raucous German student songs, and Muter, by request, gave us "The Merry Little Fat Grey Man." All the jolly old meetings, getting dim in the mist of time. . . .

The fermentation among chemists that started with the birth of the Society led, in 1877, to the foundation of the Institute of Chemistry. Muter resolutely opposed this, for what were then good reasons. The founders of the Institute were largely academicians out of sympathy with the work and the aspirations of the chemical practitioners, as the years of unrest that followed in the Institute of Chemistry clearly proved. Yet gradually both the Society of Public Analysts and the Institute got over their turbulent boyhood years and grew into lusty adultness, supporting each other and raising a bounteous crop of facts and of men—a new vigorous profession that is a power in the land, a promise for the future of England. Muter in time became a fellow of the Institute and served as Censor from 1896-1898.

Muter's journalistic energies were gradually overwhelmed by his ever-growing responsibilities, and in 1891 the Society recognised that the Journal could no longer remain under the private guidance of its honoured Past-President. It was accordingly reacquired by the Society, and has since remained under the direction of the Publication Committee, which is directly responsible to the Council.

After that time the members saw little of Muter, whose school, converted into a limited liability company, required much of his energy. He also held the appointments of Public Analyst for Bermondsey, Lambeth, Newington, Rotherhithe, Southwark, and Wandsworth—in fact, for the greater part of South London—and for part of Lincolnshire. His private practice was extensive, and included the advisorship of public bodies like the Metropolitan Asylums Board.

He died on December 19, 1911, at Worthing. OTTO HEHNER.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE Annual General Meeting of the Society was held on Wednesday evening, February 7, in the Chemical Society's Rooms, Burlington House. The President, Mr. E. W. Voelcker, A.R.S.M., F.I.C., occupied the chair.

The minutes of the previous Annual General Meeting were read and confirmed.

Mr. J. H. B. Jenkins, F.I.C., and Dr. R. Lessing were appointed scrutators of the ballot papers for election of Officers and Council for 1912.

The HON. TREASURER presented the accounts of the Society for 1911. These were adopted, and votes of thanks passed to the Hon. Treasurer, Auditors, and Hon. Secretaries.

On the motion of the PRESIDENT, a vote of thanks was passed to the President and Council of the Chemical Society for allowing the Society the use of their rooms at Burlington House during the past year.

Mr. T. Macara, F.I.C., and Mr. R. G. Grimwood, F.I.C., were appointed Auditors of the Society's accounts for 1912.

The PRESIDENT having delivered his Annual Address,

On the motion of Mr. OTTO HEHNER, F.I.C., seconded by Mr. W. J. DIBDIN, F.I.C., a vote of thanks was accorded to the President for his Address and for his services in the chair.

The PRESIDENT briefly acknowledged the vote of thanks.

The scrutators having reported the result of their examination of the ballot papers,

The PRESIDENT announced the election of Officers and Council for 1912 as follows:

President.—L. Archbutt, F.I.C.

Past-Presidents (limited by the Society's Articles of Association to Eight in Number).—Edward J. Bevan, F.I.C.; Bernard Dyer, D.Sc., F.I.C.; Thomas Fairley, F.I.C.; W. W. Fisher, M.A., F.I.C.; Otto Hehner, F.I.C.; R. R. Tatlock, F.I.C.; E. W. Voelcker, A.R.S.M., F.I.C.; J. Augustus Voelcker, M.A., B.Sc., Ph.D., F.I.C.

Vice-Presidents.—W. H. Willcox, M.D., B.Sc., M.R.C.P., F.I.C.; Arthur R. Ling, F.I.C.; John E. Stead, F.R.S., F.I.C.

Hon. Treasurer.—H. Droop Richmond, F.I.C.

Hon. Secretaries.—A. Chaston Chapman, F.I.C.; P. A. Ellis Richards, F.I.C.

Other Members of Council.—E. Richards Bolton; R. M. Clark, B.Sc., F.I.C.; J. Connah, B.Sc., F.I.C.; J. Evans, F.I.C.; Charles A. Hill, B.Sc. F.I.C.; E. Hinks, B.Sc., F.I.C.; G. T. Holloway, A.R.C.Sc., F.I.C.; G. N. Huntly, B.Sc., A.R.C.Sc., F.I.C.; G. W. Monier-Williams, B.A., Ph.D., F.I.C.; F. Wallis Stoddard, F.I.C.; S. R. Trotman, M.A., F.I.C.; S. A. Woodhead, M.Sc., F.I.C.

The monthly meeting followed the annual general meeting, the newly elected President, Mr. Archbutt, being in the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Mr. H. Hawley, M.Sc., and Miss M. Gazdar, B.Sc., A.I.C., were read for the second time; and certificates in favour of Messrs. Maurice E. Balston, M.A. (Cantab.), Buckland, Maidstone, analyst to Messrs. W. and R. Balston, paper manufacturers, Maidstone; Howard A. Caulkin, B.Sc. (Lond.), F.I.C., Mona Villa, Spencer Road, Belper, assistant chemist to the Midland Railway Company; and Charles Reginald Wilkins, B.Sc. (Lond.), 40, Church Lane, Hornsey, N., assistant to Messrs. Redwood and de Hailes, were read for the first time.

Messrs. S. G. Agar, G. S. Barton, S. W. Bridge, R. V. Briggs, F. C. Dyche-Teague, B.Sc., A.I.C., F. A. Mason, W. B. Saville, and G. A. Stokes, were elected members of the Society.

The following papers were read: "The Souring of Milk," by H. Droop Richmond, F.I.C., and H. C. Huish. "Notes on the Determination of Butter Fat and Cocoanut Oil in Margarine," by F. W. F. Arnaud, F.I.C., and H. Hawley, M.Sc.; and "A Flour Improver," by Edward Hinks, B.Sc., F.I.C.

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ANNUAL ADDRESS OF THE RETIRING PRESIDENT.

(*Delivered at the Annual General Meeting, February* 7, 1912.)

THIS evening I have to address you for the last time as President of the Society, and, until comparatively recently, I was in hopes that I should be able to announce that during the year 1911 we had not lost any of our members by death. Unfortunately, these hopes have not been realised, and I have to record the death of three very old members of the Society—Mr. McCowan, Mr. Sanford, and Dr. Muter. Mr. McCowan was a life member, who invented a new method for the manufacture of caramel. Possessed of exceedingly wide knowledge, he was at once a practical brewer and an accomplished inorganic chemist, having devised several processes for dealing with ores. Mr. Sanford was a public analyst, who had devoted much of his work to the chemistry of explosives.

By the death of Dr. Muter, a link with the past history of the Society is severed. At the time of his death Dr. Muter was the senior Past-President of the Society, having held office in 1879-80. In 1877 he was, with the late Mr. Wigner, joint-editor of the ANALYST, and on the latter's death in 1884 he became the sole editor and proprietor until the year 1890, when the Journal passed into the hands of the Society itself. Although of late years he had not kept up any active participation in the affairs of the Society, his pioneer work will always gratefully remain in the memory of his colleagues. We hope to have an interesting memoir of him from the pen of one who was intimately associated with his work.

I am glad to say that we have lost only one member by resignation, while twenty-three new members have been added to our roll, and the election of eight other new members takes place this evening. This, although not a large increase, is a satisfactory one, and our total membership is now over 400. Although necessarily our membership is restricted, there are still a number of men in official positions and in professional work whom we would welcome into our ranks, and whose inclusion would be a source of strength. At no time than now was it more necessary that the profession of analytical and consulting chemists should be drawn together in a united effort to uphold and improve the position of its members, and to resist the attempts, which are all too prevalent, of reducing our profession to the level of a competitive trading business.

In my last address I referred to the lamentable results brought about by the insane competition for official positions under the Food Acts. I am glad to say there are signs that there is a better appreciation of the mischief that is done to the whole profession by analysts agreeing to undertake such responsible work at fees incompatible with its honest performance or with decent remuneration. No one, I submit, can seriously contend that the Public Analyst's remuneration is excessive, or even liberal. It is based largely on the objectionable principle that the holder of an appointment must be content with official "sprats," so that he may have the chance of catching remunerative "whales." As we all are too painfully aware, the fee mentioned in the Act, as chargeable to individual members of the public wishing to make use of the services of the Public Analyst, has in most cases come to be the maximum fee paid by Local Authorities, in spite of the fact that the advance of knowledge has been utilised by the "scientific adulterator" in such a way as to vastly increase the labour of detection of adulteration. Another very serious attack on the position of the Public Analyst and of the unofficial analyst is revealed by the action of some County Councils in establishing laboratories of their own, where analyses are carried out either gratuitously or at purely nominal fees. The system of County Authorities appointing a "whole-time" officer for carrying out the work required by them is one which, as I stated in my previous address, has "come to stay," and it cannot be denied that there is a good deal to be said in its favour if carried out on proper lines. But the establishment of a municipal or county laboratory at the expense of the ratepayer on the lines of a municipal trading concern is highly objectionable.

In the most recent case in point a County Authority has established a Bacteriological and Chemical Laboratory under the direction of the Medical Officer of Health, for the examination of waters and the solving of questions arising in connection with sanitary matters and food-supplies. This work had previously been carried out by the Public Analyst for the county, who is thus faced with a serious loss of his professional income.

We, as the society representing the Public Analysts of the country, have strenuously fought against the principle that the work of the Public Analyst can be properly merged in that of the Medical Officer of Health. The work of the latter officer ought to be *outside* the chemical laboratory, and his knowledge and training—save in rare instances—is utterly inadequate to make it desirable that he should have control of a chemical department. Unfortunately, in the past, the powers that be did not see things in this light, but the Local Government Board has now shown that it is in sympathy with our views; and, by refusing to confirm dual appointments, has done much to discourage a practice injurious alike to our professional and to the public interests. Not only in this country, but in our overseas dominions, is this question a serious one, for I understand that it is proposed to place the Government Chemical Laboratories of one of our dominions under the control of a Medical Officer, in spite of the fact that the work of the Department consists to a large extent of metallurgical and mineral investigations.

During the past session, thirty-five papers were read at the meetings of the Society, and these—with a few exceptions—have been published in the ANALYST. It is in connection with these few exceptions that I have a serious complaint to make. Last year I appealed to members to loyally support our Society by reading at our own meetings papers on subjects in which we were closely interested. It is impossible and unreasonable to expect that papers should always be read solely with the pure love of advancing knowledge and without any desire that some personal advantage should come to the author. A hard-and-fast line, however, must be drawn at papers read for the purposes of self-advertisement, or for trade purposes. In the cases to which I refer, papers were read at our meetings and simultaneously sent for publication to Trade Journals interested in pharmaceutical matters. We, as a Society, rightly object to be "exploited" in this way, and I trust that we shall have no repetition of it.

In referring to the question of advertisements, I am glad to say that I think there is a growing feeling that the practice which, in the past, has led to so much abuse—that of giving certificates for the purposes of trade advertisement—is one unworthy and discreditable to our profession. It is very necessary that the Public Analyst should be especially careful in the matter of the reports that he gives, for, not only is he liable to be exploited by the astute advertiser under circumstances which, one must admit, give some excuse for it, but he may—as a recent instance shows—be put in the position of being compelled to take legal action to prevent the unauthorised use of his name in advertisements, or else leave himself open to the suggestion that he has, for a consideration, allowed his name to be so used. I am very glad that one of our most prominent members has had the courage and the public spirit to take legal action in order to stop such proceedings. The position of a Public Analyst would be intolerable if the reports which he has to make in the course of his official duties were liable at any time to be utilised by the pushing advertising trader. I am afraid that in some cases Public Analysts are too easily cajoled into allowing their names to be used under the plea that the matter is one of great public and national importance, and that, by so doing, they are giving valuable assistance in the cause of the health and welfare of the nation. Such popular causes as the stamping out of the great white peril—consumption—have furnished an example of how a Public Analyst may be, no doubt unwillingly, associated with a most unpleasant and unsavoury suggested treatment for this disease. The great popular movement engineered by the Press in favour of "Standard Bread," and blessed in its inception by a manifesto signed by several distinguished medical men, is a very good example of the hollowness of most of these movements. They serve the purpose for which they are started, and bring grist to the mill of the enterprising advertisers and advertising agents.

Such questions as those of sophistication of flour, the composition of baking powders, and the alleged injury which has been done to the health of our working population by the use of "white" flour instead of "standard" flour, have been investigated in the laboratory of the Local Government Board, and interesting reports have been issued which have served to place these matters further on a sounder scientific basis, and they have done much to discount the alarmist, and, in a great measure, unfounded statements of the eminent medical authorities who have allowed themselves to be so exploited.

I must again congratulate our Honorary Treasurer on the very clear financial statement which he has laid before us, and upon the careful manner in which he husbands our resources.

It will be noticed that this year we have in our accounts the item "Legal Expenses." This is an item we never like to see if it is possible to avoid it, but this year the Council felt obliged to go to some expense in opposing an application which was made by the Royal Institute of Public Health to grant that Institute a Royal Charter. In the end the Privy Council did not advise the granting of the Charter.

There are two points in connection with our expenditure which will require attention in the near future. The first is that the growing increase in the cost of our Journal is every year greater than the increase in the income of the Society from the subscriptions of new members. Our policy has been—rightly, I think—to spend as much as possible on our Journal, consistent with maintaining a proper reserve of accumulated funds. The increase in our Journal is shown by the fact that the volume for 1911 contains forty-eight more pages than that for 1910. There have been 39 Papers, occupying 128 pages, and 817 Abstracts, Reviews and Reports, occupying 484 pages in all, as against 34 Papers and 662 Abstracts in 1910. It is an increasingly difficult task to wade through the enormous mass of scientific literature which steadily accumulates, month by month, and to successfully pick out for recording in the ANALYST, only that which is good, rejecting the indifferent and bad.

The Society will, I am sure, gratefully recognise the care and zeal which our Editor, Mr. Julian Baker, shows in keeping up and improving the high standard of our Journal.

Our Research Fund—which I am glad to see has received further financial support—is the means of providing valuable matter for our Journal, and some interesting investigations have been completed and the results published in the ANALYST during the past year, while a number of other investigations are still in hand.

The second point in future expenditure to which I would refer is in connection with secretarial work. To our honorary officers, and especially to our senior Hon. Secretary, Mr. Chapman, on whom the main part of the secretarial work has fallen, the Society owes a deep debt of gratitude for the time and labour which they have so constantly and ungrudgingly devoted, gratuituously, to the affairs of the Society.

When we were a smaller Society, it was possible to cope with the secretarial work without unreasonable expenditure of time and money, but now, when the mere routine work alone involves the sending out of over 400 notices every month, I feel very strongly that it is the duty of the Society to make provision to relieve our Hon. Secretaries of a burden which has involved them in considerable annual expense.

In the coming Session of Parliament two Bills, which will be of special interest to us, are to be introduced—one, the "Milk Bill," which the President of the Local Government Board will bring forward, the other, Lord Clinton's "Sales for Agricultural Purposes" Bill, which has already passed the House of Lords. With regard to the former, the President of the Board of Agriculture has quite recently indicated what he thought should be the guiding principles which should dominate the Bill. In the first place, no milk which was likely to breed disease should be sent into towns; that farmers should not be subject to prosecution for faults over which they had no control; and, lastly, that, if prosecutions took place, they should be civil and not criminal.

The President of the Board further stated that all that the consumer had a right to expect was that he should get pure milk as it comes from the cow. Unfortunately, the attitude of the Board as to standards for pure milk is eminently unsatisfactory, and, having in 1901 issued the "Sale of Milk Regulations," which fixed the standards, they appear now to be anxious to repudiate them. In the Annual Report for 1910 of the Intelligence Division of the Board of Agriculture it was stated that the Board did not require that milk should contain 3 per cent. of fat, and that, generally speaking, it was no offence against the Food and Drugs Acts to sell milk that falls below the limits mentioned in the "Sale of Milk Regulations." If this be so, what is the use of having any Milk Regulations at all? The Board have had a great deal of pressure put upon them, especially by farmers in the North-East of England, to lower the already very low standard which has been fixed, and no doubt this is the reason of the Board's action in the matter.

When one considers the labour that was expended in connection with the milk inquiry previous to the issue of the Sale of Milk Regulations, it seems incredible that the Board of Agriculture should countenance any lowering of the standard for Milk, a course of procedure which, while, no doubt, of benefit to the producers of inferior milk, would inflict a grave injury on the public in general. To suggest that everything should be regarded as "pure milk," simply because it was what the cow had yielded, is most dangerous.

The suggestion that prosecutions in the case of milk should be removed from the criminal courts, appeals in many cases to one's sense of fairness, but I cannot see why the milk producer alone should be exempted from the odium which attaches to an appearance as a defendant in a police court. Far too much stress is being laid upon the point that the regulation of the quality of a milk-supply is beyond the control of the producer, and, while admitting that, under certain circumstances, it may be a hardship for an honest producer to be summoned in a criminal court, it must not be forgotten that it is the duty of a man who produces milk for sale to see that it complies with the very moderate requirements at present laid down. By proper selection and treatment of his cows, there should be, generally speaking, no difficulty at all in the matter.

The "Sales for Agricultural Purposes" Bill introduces several alterations to the Fertilisers and Feeding Stuffs Act of 1906, among the principal ones being the abolition of the veto of the Board of Agriculture upon prosecutions, and the extension of the time during which samples may be taken and prosecutions instituted. The principal alteration affecting analysts under the Act is that further particulars are required to be given in the invoices of articles sold for use as food for cattle or poultry. Under the proposed new Act it is suggested that the percentage of "soluble carbohydrates" shall always be stated. I think it should be pointed out that the figure for the percentage of soluble carbohydrates is only a difference item, and, to arrive at it, it is necessary to quantitatively determine five other classes of bodies in every sample examined, whereas at present the determination of two only is required in samples submitted under the Act. This should certainly call for the revision of the fees which are at present deemed to be adequate remuneration to the analyst by some of the local authorities in the kingdom. The whole tendency of the action of the authorities administrating both the "Sale of Food" and "Fertilisers and Feeding Stuffs" Acts has been to throw an increasing burden upon analysts, and even in the matter of the quarterly returns that have to be made to the Local Government Board and the Board of Agriculture additional information is constantly being required. In cases where preservatives are found in any article of food, the exact percentage is constantly asked for, and where samples of milk have been reported on as falling below the limits of the Sale of Milk Regulations, detailed particulars of the analysis are demanded.

While we are ready to place all information at our disposal in the hands of the authorities, I think it would be only fair that proper recognition should be given to the increased work which is now thrown on the Public Analyst.

With regard to the question of preservatives, I would only say that it seems extraordinary that legal effect has not yet been given to the recommendations of the Committee on Preservatives, and which were reported no less than eleven years ago. It is to be hoped that ere long a serious attempt to place our Food Acts on a really satisfactory basis will be carried out by the Government.

The proceedings of the Congress which was held in 1910 in Paris, at which we were represented by our Hon. Secretary, Mr. Chapman, have been published recently in the ANALYST, and I understand that, in all probability, a further Conference to discuss the question of the Uniformity of Methods of Analysis of Foodstuffs will be held.

I have now to relinquish this chair, and, in introducing my successor, Mr. Archbutt, I heartily congratulate the Society upon having secured the services of one who has been so long closely associated with and interested in our affairs. Mr. Archbutt is, as you all know, a distinguished chemist, with a very wide field of knowledge and experience. As chemist to one of our great railway companies, he has distinguished himself by many important investigations on subjects of vast industrial importance, and, incidentally, although he is not a Public Analyst, his services have been largely requisitioned in connection with work which belongs to this branch of the profession. Mr. Archbutt has the distinction of being the first President of this Society who has, I believe, never held the post of Public Analyst, and I think that it was a very happy suggestion that the gradual enlargement of our horizon—which a few years ago resulted in our incorporation as a Society which embraced all branches of the analytical profession—should now be marked by the election of Mr. Archbutt to the presidential chair.

I now come to the most difficult part of my address, and that is to adequately express to you how deeply I appreciate the honour that has been conferred upon me in placing me in this chair for two years. I vacate it with the greatest possible regret, but I shall always look back with pleasure on the period that I was permitted to occupy it. From the general body of members I have received nothing but kindness, and I cannot sufficiently thank my colleagues on the Council—and especially the honorary officers—for their help and support always cordially given me.

Though relegated to the list of "have beens," I shall always continue to carry out what has been my great object throughout my official connection with the Society—that is, to do everything I can to promote the welfare and position of the Society and of our profession.

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THE ESTIMATION OF SMALL QUANTITIES OF ESSENTIAL OILS IN SPICES, ETC. PART II.

BY J. A. BROWN, F.I.C.

(*Read at the Meeting, December* 6, 1911.)

IN a previous paper (ANALYST, 1910, **34**, 392) I described a dry-distillation-combustion process for estimating the essential oils in carraway and cinnamon spices. The method has now been applied to some other essential oils with the following results:

	Carbon Value.	Temperature of Volatilisation.	Average Time of Experiment.	Correction.	Essential Oil in Spice.
					Per Cent.
Coriander ...	75	140° C.	½ hour.	None.	—
Cardamom ...	73	140° C.	½ „	„	1·02
Rosemary ...	79·5	140° C.	½ „	„	0·60
Eucalyptus ...	74 73 } 73 72	140° C.	¾ „	„	1·40
Aniseed ...	77	150° C.	1 hour.	Deduct 0·1 per cent. oil for every ½ hour's heating.	{1·00 {2·20
Bay	76	150° C.	½ „	Deduct 0·1 per cent. oil for every ½ hour's heating.	0·75
Juniper ...	83·5	150° C.	¾ hour.	Add one-sixth oil found, and deduct 0·15 per cent. for ¾ hour's heating.	1·30
Cedar... ...	83 84 } 83·5	160° C.	¾ hour.	Add one-eighth oil found, and deduct 0·25 per cent. for ¾ hour's heating.	2·85
Santalwood ...	82	160° C.	—	Add one-sixth oil found.	—
Origani ...	79·5	—	—	—	—
Black pepper	85	—	—	—	—
Pimento ...	73	—	—	—	—

EUCALYPTUS.—In the case of three oils from different sources the carbon values were obtained lower than would be expected on theoretical grounds supposing the composition to be mainly terpenes and eucalyptol. The third result given was from a sample of oil guaranteed to be from *Eucalyptus globulus*.

ANISEED and BAY require a deduction to be made from the percentage of oil found, to allow for a slight amount of destructive distillation. When spices have to be heated above 140° C. to volatilise the essential oil, a small allowance has to be made for destructive distillation. Santalwood appears to be the only exception.

CEDAR, JUNIPER, and SANTALWOOD, containing more difficultly volatile oils, require a temperature of 150° to 160° C. There is a tendency to resinify under the conditions of the process. Thus, oil of cedar showed unburnt residues of 12·6 and 13 per cent., and santalwood 16 per cent.

Juniper berries showed a similarity to carraway-seed (*loc. cit.*) in that, after drying at 100° C. until constant in weight, further oil—viz., 0·50 and 0·72 per cent.—was obtained on combustion. A sample of spent berries supplied to me by a distiller gave 0·15 per cent., which proves the comparative inefficiency of ordinary laboratory methods compared to those of the distiller who uses superheated steam.

GINGER.—After repeated trials, the method had to be abandoned, owing to the ease with which this oil resinified.

It is hoped to apply the process to the estimation of nicotine in tobacco and oil of mustard in mustard. I have to thank Messrs. Stafford, Allen and Sons, and Mr. J. C. Umney for supplying me with samples of oils, etc., and also Dr. Woollatt for help and advice.

DISCUSSION.

Mr. E. J. PARRY considered that this method was open to criticism, on account of the large variations which occurred in the constituents of many essential oils. For instance, in the eucalyptus oil of the British Pharmacopœia, the proportion of eucalyptol was liable to vary at different times. There was rarely less than 50 per cent., but most of the genuine oil of the present year contained 75 or 80 per cent. About thirty constituents had been isolated from various eucalyptus oils. The terpenes would contain about 88 or 89 per cent. of carbon, and the less oxygenated constituents from 78 to 80 per cent., so that one might have 75 to 80 per cent. of eucalyptol with a carbon value of 88 or 89 per cent., and the remainder with a carbon value varying from 78 or 79 to 88 or 89 per cent. The result was that a carbon figure of, say, 72, 73 or 74 might mean almost anything. The position was exactly the same with regard to bay oil, rosemary oil, and many others.

Mr. A. CHASTON CHAPMAN remarked that Mr. Brown's process was in reality one for determining certain volatile constituents of spices—not necessarily the actual proportion of essential oils present. Bay oil, for example, to which allusion had been made, underwent considerable resinification on exposure to air, and this would, of course, very considerably diminish the proportion of volatile constituents obtained by the process. There could be no doubt that for certain purposes the method was a very useful one, but it seemed to him (Mr. Chapman) that it would be very dangerous to attempt to put it forward as a general method for distinguishing between genuine and exhausted spices, since there might be little or no difference between an exhausted sample and a sample in which a considerable proportion of the constituents of the essential oil had become converted into non-volatile matters by exposure to the air.

Mr. C. A. Hill remarked that to speak of "eucalyptus oil" was not definite, and drew attention to the variation in the proportion of eucalyptol. In Tasmanian (globulous) oil it was usually from 55 to 60 per cent., whilst in Australian oil it was often as much as 80 per cent.

Mr. H. E. Burgess said that he had found that, by extracting the spice with petroleum ether, and determining the refractive index, a very good indication was obtained of the quantity of essential oil present. All analytical methods for this purpose were, however, liable to such errors as had been mentioned, and probably the only way in which a true indication of the percentage of essential oil could be obtained was by actually distilling a considerable quantity under manufacturing conditions.

The Chairman (Mr. Ling) remarked that from the discussion it appeared that the weak point in the method was the application of a constant factor derived from what was supposed to be a typical oil to an extract that varied in composition according to the age of the spice. He should like to ask if Mr. Brown had tried heating the spice under reduced pressure. In this way resinification might to some extent be prevented.

Mr. Brown said that he had found combustion under reduced pressure to be of very little advantage—not sufficient to compensate for the additional trouble and difficulty of manipulation which it involved. With regard to Mr. Parry's remarks, he would point out that the estimations in the spices were made under exactly the same conditions under which the carbon figures were obtained, so that whatever error there might be would be the same in each case. As he had mentioned in the previous paper (Analyst, 1910, **34**, 392), the carbon figure did not seem to be affected to any great extent by variations in the proportions of the different constituents of the oil, and, in cases in which the proportion of essential oil was small, such differences would, he thought, not be material. Of course, however, it was not suggested that the method gave more than a fairly close approximation to the truth.

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A FLOUR IMPROVER.

By E. HINKS, B.Sc., F.I.C.

(*Read at the Meeting, February* 1, 1912.)

An article forming a recent addition to the number of substances sold as flour improvers consists of potassium persulphate. According to the directions it is to be added to flour in the proportion of from ½ to 1 ounce to the sack of flour—*i.e.*, about 1 of persulphate to from 10,000 to 5,000 parts of flour. The dry persulphate is simply mixed with the flour.

It is claimed that by such treatment stronger loaves and loaves of better colour are obtained, more water can be absorbed in the doughing process, and, further, that the only residuum in the loaf as a result of the treatment is a minute proportion of a harmless salt, which is a normal constituent of wheat and of many drinking waters.

The presence of persulphate in the flour may be readily detected by the reaction with benzidine (Rothenfusser, ANALYST, 1909, **34**, 37). An alcoholic solution of benzidine gives a blue colour with extremely dilute solutions of persulphate, the limit of delicacy being 1 part in about 1,000,000 parts of water; but on applying this test to aqueous extracts of flour the reaction is much less delicate, and an extract of flour containing 1 part of persulphate in 5,000 parts of flour (the greater strength recommended), fails to give the reaction. Much smaller quantities than this, however, may be detected by making a paste of the flour with a small quantity of water and at once pouring the benzidine solution over the paste. The benzidine picks out the particles of persulphate and develops the blue colour round each particle. By proceeding in this manner, flour containing persulphate in the proportion as recommended gives strong reactions, and the limit of delicacy depends practically only on the quantity of the flour taken. With laboratory preparations carefully mixed and sieved, 5 to 10 grms. of flour with 1 of persulphate to 100,000 will, as a rule, show the reaction. A convenient benzidine reagent is a 2 to 3 per cent. alcoholic solution.

Mixtures of persulphate with flour gave the reaction with no apparent diminution of intensity after standing for three months.

The persulphate may be separated from the flour by suspending in carbon tetrachloride and centrifugal action, the benzidine being applied to an aqueous solution of the deposit. Further, the salt can be quantitatively determined by centrifugalising the suspension of flour in carbon tetrachloride, treating the deposits with small quantities of water and again centrifugalising or filtering to remove small quantities of flour unavoidably left in the tubes, warming with potassium iodide and titrating with thiosulphate in the usual manner.

In the process of baking the persulphate would appear to be entirely destroyed. Loaves were made from several mixtures, but even when the persulphate was present to the extent of 1 to 500 of flour, no benzidine reaction could be obtained on treatment of the finished loaf.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Bitter Principles in Wort and Beer. Neumann. (*Wochensch. Brau.*, 1912, **29**, 31-36.)—Wiegmann (*Allg. Brauer- u. Hopfenzeit.*, 1911) has described a method for extracting the bitter resins from hopped worts and beer. The difficulty with the usual solvents of low boiling-point lies in the formation of emulsions which do not separate completely. Wiegmann therefore recommends the use of petroleum spirit of sp. gr. 0·7350, so that the emulsion which tends to be formed may be destroyed by heating subsequently in a water-bath at 80° to 85° C.; 500 c.c. of the wort are shaken with 100 c.c. of petroleum spirit, and transferred to an Erlenmeyer flask of 1 litre capacity, which is placed in the water-bath. The flask is connected with a condenser, by means of a short piece of rubber tubing, in such a way that the contents of the flask can be shaken vigorously in the water-bath until the emulsion is destroyed. The liquid is then poured carefully into a separating funnel, the wort is removed, the petroleum spirit is washed, filtered, treated with alcohol, and the dissolved bitter principles are titrated with alcoholic potassium hydroxide. Some beers give trouble even under the above conditions, but emulsions can generally be prevented if the sample be first boiled with 1 to 2 per cent. of phosphoric acid (sp. gr. 1·12) for about fifteen minutes under a reflux condenser.

The author describes a simpler method than that recommended by Wiegmann for overcoming the emulsion difficulty. Ether is used as the extracting solvent, and the difficulty is overcome by employing an unusually large proportion of ether, and extracting the beer in small portions at a time. Fifty or 100 c.c. of flat beer are shaken with five times their volume of ether in a long-shaped separating funnel, preferably in a shaking machine. After drawing off the extracted beer, a further quantity of 50 or 100 c.c. of the beer are treated with the same lot of ether, and so on until the whole of the sample has been exhausted. No difficulty in separating is experienced, even with dark beers.

J. F. B.

Estimation of Benzoic Acid in Margarine, Butter, and other Fats. W. Friese. (*Pharm. Zentralhalle*, 1911, **52**, 1201-1203; through *Chem. Zentralbl.*, 1912, **1**, 96.)—One hundred grms. of the sample are thoroughly mixed with 25 c.c. of 20 per cent. sodium carbonate solution, and heated on the water-bath until a milky fluid separates beneath the fat. The beaker is then cooled in an ice chest, and the layer of fat removed and washed repeatedly with ice water. The aqueous layer and washings, which will contain all the benzoic acid as sodium benzoate, are treated, without filtration and in the cold, with 40 c.c. of 10 per cent. barium chloride solution, which will precipitate barium salts of the saponified fat and barium carbonate. The liquid is filtered, and the filtrate freed from barium by means of sulphuric acid and again filtered. The new filtrate is twice extracted with ether in a separating funnel, the united ethereal extracts washed with water and dried over calcium chloride, the solvent evaporated by means of a current of air, and the residue dried in a desiccator and weighed. C. A. M.

Estimation of Camphor. H. C. Fuller. (U.S. Department of Agriculture, Circular No. 77, Bureau Chem.; through *Amer. J. Pharm.*, 1912, **84**, 40-41.)—The author employs the camphoroxime method for determining camphor in its preparations, and the following outline illustrates its application to spirit of camphor: Exactly 35 c.c. of a hydroxylamine solution (containing 20 grms. hydroxylamine hydrochloride, 30 c.c. water and 125 c.c. of aldehyde-free absolute alcohol) are added to a mixture of 25 c.c. of the sample with 2 grms. of sodium bicarbonate, and the whole gently boiled for two hours under a reflux condenser. On cooling, 6 c.c. of hydrochloric acid (sp. gr. 1·12) and 6 c.c. of water are added, the condenser and flask washed out, the liquid diluted to 500 c.c., and filtered. Fifty c.c. of the filtrate are neutralised with alkali to methyl-orange (mineral acid), when phenolphthaleïn is added and the excess of hydroxylamine titrated with $\frac{N}{10}$ alkali. A blank experiment, using 35 c.c. hydroxylamine solution and 25 c.c. alcohol, is made, and the difference in the titrations noted. Each c.c. of $\frac{N}{10}$ alkali$=$0·01509 grm. camphor.

A. R. T.

Estimation of Cinnamein in Peru Balsam. F. Lehmann and A. Müller. (*Arch. Pharm.*, 1912, **250**, 1-5.)—The method of estimating cinnamein described in the German Pharmacopœia is shown to be inaccurate owing to no account being taken of the increase in volume in the ether used for the extraction. This may cause an error of more than 1 per cent. in the estimation. The following process based on those employed by Fromme and by Stocker is recommended as accurate and rapid. The use of so large a quantity of tragacanth as 3 grms. (Fromme) is unnecessary, provided that the bulk of the water be first drawn off, leaving only a small residue to be absorbed. Two and a half grms. of the Peru balsam are shaken with 5 c.c. of water in a stoppered 75 c.c. flask, after which 30 grms. of ether are added, and the shaking continued for one minute. An addition of 5 grms. of sodium hydroxide solution is now made, and the flask once more shaken for one minute and allowed to stand for ten minutes. It is next inverted, the stopper partially withdrawn, and the aqueous layer drawn off down to about 3 c.c., after which 0·5 grm. of gum tragacanth is introduced, and the whole thoroughly shaken and allowed to stand for three to five minutes. The bulk of the ethereal layer (25 to 28 grms.) is now decanted into a wide-mouthed tared flask, which is immediately covered and weighed. The ether is evaporated on the water-bath, and the viscous residue of cinnamein dried for thirty to forty-five minutes at 100° C., cooled in a desiccator, and weighed. C. A. M.

Detection of Cocoanut Oil in Butter and Lard. E. Polenske. (*Arbeit. Kaiserl. Gesundheitsamte*, 1911, **38**, 402-414.)—The results of a critical examination of the methods proposed by Ewers (*cf.* ANALYST, 1910, **35**, 353) and Fendler (*ibid.*, 1910, **35**, 354) are recorded. Genuine butters of known origin were employed in the experiments, and it was found that the methods mentioned cannot be relied on for the purpose of detecting the presence of cocoanut oil in butter fat. As regards

Ewers' method, butter fats having Reichert-Meissl values of from 29·9 to 31·2 yielded "difference values" varying from 8·5 to 8·8; other samples, also having high Reichert-Meissl values, gave "difference values" of from 13 to 14, and these samples could be adulterated with 10 per cent. of cocoanut oil and still show "difference values" above the minimum limit of 9 fixed by Ewers.

Fendler's alcohol method (*loc. cit.*), when applied to genuine butters, yielded values varying from 28·7 to 31, whilst several butters gave from 9 to 11 c.c. of ethyl esters when examined by the ester method (*loc. cit.*).

The Polenske value of the butters used in the above investigation were normal, and showed that this method is trustworthy; further evidence is adduced that the method is also reliable for the detection of cocoanut in lard. W. P. S.

Fruit Juice Statistics for the Year 1911. A. Behre and K. Frerichs. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 733-736.)—Juices prepared from fermented fruit and examined during the year yielded the following results: *Strawberry juice* (5 samples): alcohol, 0·44—2·39; total solids, 3·49—4·78; acidity (as malic acid), 1·19—1·75; ash, 0·37—0·52. *Raspberry juice* (5 samples): alcohol, 0·30—2·26; total solids, 7·06—9·61; acidity, 3·35—4·32; ash, 0·75—0·89. *Currant juice* (6 samples): alcohol, 0—2·32; total solids, 5·73—6·74; acidity, 2·76—3·79; ash, 0·61—0·74. *Pine-apple juice* (4 samples): alcohol, 0—0·53; total solids, 7·08—16·28; acidity (as citric acid) 0·60—1·28; ash, 0·35—0·67; invert sugar, 4·03—7·15; sucrose, 0—8·33. The above figures express grms. per 100 c.c. As regards the pine-apple juices, two of the samples were unfermented, and two slightly fermented; the sucrose was completely inverted in the fermented samples. Two of the samples of currant juice yielded a positive reaction with Windisch's test for cherry juice (*cf.* ANALYST, 1902, **27**, 10); the coloration obtained was very slight, and the authors are unable to explain why the juice should contain hydrocyanic acid. W. P. S.

Occurrence of Arsenic in Gelatin. O. Köpke. (*Arbeit. Kaiserl. Gesundheitsamte*, 1911, **38**, 290-293.)—Of twelve samples of commercial gelatin examined by the author, no less than eight contained appreciable quantities of arsenic, the amounts varying from 0·05 mgrm. to 0·3 mgrm. per 10 grms. of the sample. The remaining four samples also contained arsenic, but the quantity was a mere trace. With regard to the origin of the arsenic, it is pointed out that gelatin is prepared from hide and leather residues, the hides having been treated with lime and arsenical preparations, or from bones which have been extracted with impure mineral acids, etc.; gelatin is also bleached with sulphurous acid, which is not always free from arsenic. W. P. S.

Estimation of Gum in Syrups. A. C. Chauvin. (*Ann. Falsif.*, 1912, **5**, 27-30.)—The usual method for the estimation of gum by precipitation with an alcoholic solution of lead acetate gives satisfactory results when certain precautions are observed. The author, however, prefers to precipitate the gum-acid by means of alcoholic hydrochloric acid. For instance: 5 c.c. of the solution are treated with 5 c.c. of hydrochloric acid, 5 c.c. of water, and 85 c.c. of 100 per cent. alcohol. It is particularly important to add the reagents, both acid and alcohol, in a rapid stream of drops, but not as a continuous jet, and to keep up a rotatory movement of the gum solution whilst these are being added. The precipitate is filtered off, washed with 95 per cent. alcohol, dried and weighed, and the dry weight is increased by the percentage of ash and moisture in the variety of gum present, or alternatively by 16·1 per cent., representing average values of 2·4 per cent. for ash and 13·7 per cent. for moisture. The quantity of sugar present does not interfere with the results provided the precipitate has been thoroughly washed, but pure aqueous solutions of gum nearly always give more accurate results. Ten per cent. solutions of gum generally show slightly low results, whilst 20 per cent. solutions show values ranging from 19 to 21 per cent. J. F. B.

Mutarotation of Honey. L. Rosenthaler. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 644-647.)—In order to determine the mutarotation of honey, 10 grms. of the sample were dissolved in water, with the addition of alumina cream, made up to 100 c.c., and the solution filtered. The solution of the filtrate was then observed in a 200 mm. tube fifteen minutes after the water has been added. Another similar solution was prepared and treated with ammonia gas, the solution being observed at the end of thirty minutes. The difference between the first rotation, *a*, and the second, *e*, gives the mutarotation, *m*. For purposes of comparison, the latter value may be calculated into the specific mutarotation—$[m] = \frac{100\,m}{l \times c}$, where *l* is the length of the tube and *c* the total solids of the honey solution. As the result of the examination of forty-one samples of honey, it was found that for normal honey the value *m* varied from 0·12 to 2·60, and that of [*m*] from 0·71 to 15·47. All the samples of honey which had become solid (grained) exhibited a high mutarotation, and, assuming that dextrose exists in two modifications, it appears that crystallisation is accompanied by a change of the less optically active modification into the more strongly rotatory modification. Invert sugar showed a specific mutarotation of 15·08, whilst the value in the case of two samples of starch syrup observed was 1·20 and 2·60, respectively. W. P. S.

Estimation of Mustard Oils in Oil-Cakes and Mustard Flours. C. Brioux. (*Ann. chim. anal.*, 1912, **17**, 3-9.)—In the author's experience the method of oxidising the essential oils with bromine and estimating the sulphuric acid produced is unreliable, the oxidation being only partial. The method based upon the decomposition of the essential oil by means of silver nitrate is more accurate, but the modification prescribed by the French Codex gives results about 10 per cent. too low. The following method of estimation is suggested: A mixture of 25 grms. of the finely ground oil-cake with 500 c.c. of water at about 37° C., and 2 grms. of sodium fluoride (previously dissolved in a little water) as an antiseptic, is kept for an hour in a water-bath at 37° to 40° C., the closed flask being shaken from time to time. In the case of certain foreign oil-cakes or of mustard flour, it is necessary to continue the maceration for three or four hours, or longer. After the enzymic action is complete, 20 to 25 c.c. of 95 per cent. alcohol are added, the contents of the flask distilled, and 150 c.c. of the distillate collected in a 250 c.c. flask containing 10 c.c. of concentrated ammonia solution. From 25 to 50 c.c. of $\frac{N}{10}$ silver nitrate solution (according to the probable quantity of essential oil) are now added, and the flask heated for an hour beneath a reflux condenser, on a water-bath at 80° to 85° C., after which the liquid is made up to 250 c.c. and filtered. An aliquot portion (125 c.c.) of the filtrate is acidified with 10 c.c. of strong nitric acid, and treated with 10 c.c. of a 10 per cent. solution of iron ammonium sulphate, and the residual silver titrated with $\frac{N}{10}$ ammonium thiocyanate solution. In the case of colza or rape-seed oil-cakes the essential oil consists of crotonyl thiocyanate, whilst in the case of black mustard-seed oil-cake or flour, the oil is allyl isothiocyanate. The distillate from Indian colza oil-cakes usually contains a mixture of the two essential oils, since the oil-cakes are composed of mixtures in varying proportions of *Brassica glauca*, *B. dichotoma*, *B. ramosa* and *B. campestris* with more or less *B. juncea* (Sarepta mustard). Of these seeds only *B. juncea* yields allyl thiocyanate, whilst the others produce crotonyl thiocyanate. Indian colza oil-cakes containing too great a proportion of the seed of *B. juncea* or *B. nigra* are thus dangerous to cattle. Apart from a microscopical examination, it is possible to make an approximate estimation of the amount of these seeds from the relative proportions of the two essential oils. For this purpose the liquid, after completion of enzymic action, is distilled without the addition of alcohol, and the distillate collected in the absence of ammonia. Crotonyl thiocyanate (sp. gr. 0·984) will then collect upon the surface of the liquid, whilst allyl thiocyanate (sp. gr. 1·028) will form a layer at the bottom. As a rule the temperatures employed in modern oil-factories are too low to inhibit completely the activity of the myrosin. French colza and rape-seed may yield from 0·150 to 0·250 per cent. of crotonyl thiocyanate, and their oil-cakes generally give from 0·20 to 0·35 per cent. In Danubian colza oil-cakes the author has obtained by the method described from 0·50 to 0·70 per cent. of essential oil, whilst Indian colza oil-cakes have yielded from 0·640 to 0·858 per cent. calculated as crotonyl thiocyanate, but generally containing a certain proportion of allyl thiocyanate. C. A. M.

Wine Statistics for the Year 1909-1910. A. Gunther. (*Arbeit. Kaiserl. Gesundheitsamte*, 1911, **39**, 1-470.)—The results of the analyses of 802 samples of wine and 3,991 samples of must are recorded in the present report, the samples being classified for the different vine-growing districts. As in previous reports (*cf.* ANALYST, 1910, **35**, 64, 526), the proceedings of the Official Commission on Wine Statistics are given. The variation in acidity of wine during storage has been further investigated, and experiments have been carried out regarding fermentation in the presence of ammonium compounds, the alteration undergone by wine solids during drying, the effect of neutralising or diminishing the acidity of wine by the addition of calcium carbonate, etc. With respect to the use of nicotine solutions as insecticides on vines, one investigator states that nicotine cannot be detected, either chemically or by the taste, in wine made from vines so treated, whilst another reports that nicotine could be tasted distinctly in wine made two weeks after the vines had been sprayed. The iodometric method described by Zeissl and Fanto (*cf.* ANALYST, 1909, **34**, 399) for the estimation of glycerol in wine was found to yield trustworthy results. The presence of large quantities of sugar was without influence on the results except that mannose, when present, caused the figures to be too high. The ordinary lime method gave low results when applied to wines containing much sugar, only about 75 per cent. of the glycerol present being obtained. W. P. S.

Composition of Guadaloupe Rum. J. Sanarens. (*Ann. Falsif.*, 1911, **4**, 642-644.)—The following results were obtained on the analysis of three samples of rum imported into France from Guadaloupe. The samples were taken as the spirit was discharged from the vessel, and the figures express parts per 100,000 of absolute alcohol. The maximum and minimum limits fixed by the French authorities for this class of spirit are also given:

	No. 1.	No. 2.	No. 3.	Limits.	
				Minimum.	Maximum.
Volatile acids ...	71·25	67·90	42·78	—	—
Total acids ...	148·99	140·93	77·79	80	150
Aldehydes... ...	13·92	20·13	14·58	15	30
Esters	47·14	67·78	64·82	50	140
Higher alcohols ...	100·67	92·28	120·09	120	200
Furfural	1·17	1·84	0·64	1	3
Total ...	311·89	322·96	277·92	300	450

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Amyloclastic Secretory Capacities of the Embryo and Aleurone Layer of Hordeum. F. Stoward. (*Ann. Bot.*, 1911, **25**, 799-841, 1147-1204.)—A North African and a brewing Chilian barley were used in this investigation, the seeds being sterilised either with 10 per cent. copper sulphate solution or absolute alcohol prior to dissection. The medium which served as the basis of the culture experiments, and to which were added as required gelatin, sugars, agar, asparagine, etc., had the following composition per 100 c.c. of solution: Calcium sulphate 0·1 grm., potassium chloride 0·25 grm., magnesium sulphate 0·25 grm., potassium dihydrogen phosphate 0·025 grm., and 2 drops of $\frac{N}{5}$ ferric chloride to the litre. The enzyme activity is expressed in terms of mgrms. of copper per twenty objects per hour, "objects" being either inner endosperms, embryos, or aleurone layers, and the copper being that obtained from Fehling's solution as the result of the action of reducing sugar formed from starch during the experiment. The copper reductions were for the most part carried out by Bertrand's method (*Bull. Soc. Chim.*, 1906, **35**, 1285).

A large number of digestions under varying conditions are described, designed to throw light on the amylase secretory capacity of the barleycorn, the following being some of the principal conclusions deduced:

Both embryos and aleurone layers possess an amylo and a cytoclastic secretory function. These secretory functions are completely arrested and annulled when these objects are subjected to the action of anæsthetic reagents such as chloroform and nitrobenzene. This result, in the case of the aleurone layer, is regarded as evidence that this tissue possesses vitality.

The inner endosperm, under varying conditions of experiment described, possesses the capacity of augmenting its amylase content, not however by secretion, a function that the amyliferous cells do not possess. In contradistinction to the secretory capacities of the embryo and aleurone layer, the augmentation of amylase is not arrested or diminished by the action of chloroform or nitrobenzene. This latter fact is taken to signify that (1) the inner endosperm represents a mass of non-living tissue, and that (2) its behaviour under the conditions described is such as a dead mass of enzyme-containing tissue would exhibit.

Isolated endosperms possess the capacity of inducing complete auto-digestion and auto-depletion of their storage contents. Isolated inner endosperms are incapable of inducing auto-digestive and auto-depletive processes in any way comparable with those observed in the case of endosperms.

The augmentation of amylase by the endosperm in presence of anæsthetics such as chloroform and nitrobenzene undergoes marked reduction, being reduced to the same order of magnitude as that of the inner endosperm. The augmentation of amylase by the inner endosperm is unaffected—*i.e.*, it is of the same order of magnitude as that of non-anæsthetised inner endosperms. Anæsthetised endosperms, unlike non-anæsthetised endosperms, are capable of inducing complete auto-depletion of their reserve storage materials.

Endospermic depletion, as it occurs in the intact seed, is principally conditioned by the action of enzymes secreted by the aleurone layer and embryo; in the isolated endosperm, by the aleurone layer secretions; and in both cases the principal rôle is due to the aleurone layer; the amyloclastic enzyme pre-existent in the inner endosperm and that which it generates is adjunctive.

The amyloclastic enzymes secreted by the embryo and aleurone layer are identical, and are distinct from that existent in and generated by the inner endosperm.

Sucrose inhibits the secretion of amylase by the embryo, dextrose diminishes it, but invert sugar has apparently no influence on the phenomenon.

The suggestion that the amylase which arises as secretions of the embryo and aleurone layer may activate that of the inner endosperm is not supported by experimental evidence. H. F. E. H.

Estimation of Colloids in Arable Soils. J. König, J. Hasenbäumer, and C. Hassler. (*Landw. Vers-Stat.*, 1911, **75**, 377-441.)—The mineral and organic colloids are important factors of the fertility of a soil since they absorb and retain the soluble nutrient salts useful to the plant. A measure of the richness of a soil in colloidal constituents may be obtained by estimations of the absorption-capacity of the soil towards dyestuffs or mineral salts. A suitable dyestuff is methyl violet in aqueous solution at 1, 2 or 3 grms. per litre, according to the capacity of the soil. Five grms. of the soil are shaken with 100 c.c. of the dyestuff solution, and after thorough clarification the amount of dyestuff removed by the soil is estimated colorimetrically. The absorption of the dyestuff depends chiefly on the colloidal clay constituents of the soil. For estimating the adsorption capacity of the soil towards mineral salts, a $\frac{N}{50}$ solution of dipotassium phosphate is used, the base and acid of which are absorbed by the soil in proportions approximately the same as they exist in the salt. The potash, however, is retained mainly by the clay colloids in a state of true adsorption which is readily reversed by treatments which flocculate the colloids; the phosphoric acid, on the other hand, is taken up mostly by the lime and ferric oxide of the soil in chemical combination forming insoluble phosphates. If the soil be dried with the phosphate solution and then washed with a definite amount of water, the quantity of potash absorbed is lower and the quantity of phosphoric acid is higher than when the soil is not dried with the salt solution. For the estimation of absorbed or adsorbed ions in soils, whether natural or previously treated with nutrient solutions as above, the authors liberate these ions by flocculation or oxidation of the colloids. Two methods for effecting this liberation have already been described—viz., by steaming and by oxidation with hydrogen peroxide. A new method is now recommended which consists in treating the soil with a powerful continuous electric current. The apparatus used is a dialysing cylinder, open at the top and closed by parchment at the bottom, in which are placed 200 grms. of soil stirred to a sludge with water, and with a layer of about 4 cm. of water on the top. This is suspended in the container, and the water outside the dialyser is filled up to the same level as inside. Platinum electrodes are arranged so that the anode is at the bottom of the outer vessel and the cathode is immersed in the dialysing cylinder. A tension of 220 volts is employed, the resistance being regulated so that the current does not exceed 3 ampères. The liquid in the inner and outer vessels is renewed when the temperature rises to 50° C. The liquid in the cathode chamber becomes alkaline, whilst that in the anode chamber becomes acid and contains humus matters; the permanganate values of the two liquids afford an indication of the organic matters, and the liquids are then mixed in aliquot proportions for the estimation o total solids, mineral matter, potash, lime, and phosphoric acid. Exhaustive treatment by the electrical method generally affords results very much higher than those obtained by steaming or oxidation. The authors therefore prefer to extract the soil twice by steeping with water for ten to twelve hours and then to subject it only once to the electrical treatment until the temperature rises to 55° C. The washings and the electrical dialysates are then united for analysis. By this treatment the results for potash coincide fairly closely with those afforded by the steaming and oxidation methods, and for phosphoric acid the single electrical and the steaming methods agree fairly well. Relations between the absorption capacity for dyestuffs or for dipotassium phosphate and the crop-yield exist in the case of the upper and lower portions of the same soil but do not hold for different kinds of soil; nevertheless, the dyestuff test can be recommended as a simple and rapid preliminary test for the general character of the soils. Comparisons between the amount of nutrient ions assimilated by the crop and the results found by the three analytical methods mentioned showed good concordance only for the potash. As regards the phosphoric acid, the steaming and single electrical treatments generally gave two to five times the amount assimilated by the crop; the oxidation method only showed relationships in the case of upper and lower portions of the same soil, since the phosphoric acid results by this method depend on the quantity of humus colloids present. For the lime, the results for all three methods were far in excess of the amount required by the plant. J. F. B.

Modification of Riegler's Method for the Estimation of Urea in Urine. T. Ekecranyz and K. A. Sodermann. (*Zeitsch. physiol. Chem.*, 1912, **76**, 171-176.) —Reigler's method, which depends upon the fact that urea is decomposed into equal volumes of carbon dioxide and nitrogen when heated with nitric acid containing a small quantity of nitrous acid, may be combined with Dumas' method for the estimation of nitrogen in organic substances. The combined method consists in measuring the volume of nitrogen liberated from a given volume of urine when the latter is heated with a modified form of Millon's reagent. One c.c. of the urine is placed in a test-tube which is closed with a cork through which pass an inlet tube, a delivery tube, and the stem of a tapped funnel. The delivery tube is connected with the lower end of a nitrometer filled with 50 per cent. potassium hydroxide solution, and a current of carbon dioxide free from air is then passed through the test-tube until the complete absorption of the bubbles in the nitrometer shows that the apparatus is filled with carbon dioxide. The level of the potassium hydroxide solution in the nitrometer is then adjusted, 2 c.c. of the modified Millon's reagent are admitted to the test-tube from the tapped funnel, and the contents of the latter are boiled for a few minutes, the gases evolved being driven over into the nitrometer by means of a current of carbon dioxide. When nitrogen ceases to collect, its volume is read off, corrected to normal temperature and pressure, and calculated into urea.

W. P. S.

ORGANIC ANALYSIS.

Estimation and Elimination of Sulphur Compounds in Commercial Benzol. J. G. Ellerton. (*J. Soc. Chem. Ind.*, 1912, **31**, 10-12.)—The principal sulphur compounds occurring in benzol are carbon disulphide and thiophene. So large an amount as 20 per cent. of the former compound may be present without imparting its peculiar odour to the benzol.

The Carius method of determining sulphur in organic compounds is here inapplicable, owing to the small content of sulphur. Garrett and Lomax's method (ANALYST, 1906, **31**, 52) for sulphur in petroleum gives low results in the case of benzol, owing to the volatility of the latter. Irwin's method, generally accepted for the estimation of sulphur in benzol, is an adaptation of the well-known "Gas Referee's Test." It gives concordant but low results, as Irwin pointed out. The author finds that a factor approximating to 1·8 will give a fairly good result, as shown by the following figures:

Sulphur Present. Grains per Gallon.		Sulphur Found. Grains per Gallon.		Factor.
486	...	277	...	1·75
486	...	283	...	1·72
486	...	270	...	1·80
564	...	307	...	1·83

For the estimation of *carbon disulphide* only, Macagno's modification of Nickel's method is used. The benzol is agitated with alcohol potash, and the potassium xanthate so formed titrated with $\frac{N}{10}$ copper sulphate solution, using potassium ferrocyanide as indicator. An approximation may be made from the density of the benzol by subtracting from it the value 0·885, and dividing by 0·00346.

Thiophene is determined by Denigès' method, a strong solution of mercuric sulphate being added to the benzol dissolved in methyl alcohol, and the compound formed dried at 115° C., and weighed. Carbon disulphide masks the reaction and interferes slightly, but the method is useful for all ordinary benzols containing 1 per cent. or less of carbon disulphide.

Owing to its different boiling-point, carbon disulphide is easily removed from benzol. This is not the case with thiophene, however, which is usually washed out by means of concentrated sulphuric acid, a method entailing the loss of about 8 to 12 per cent. of benzol. The best results for removal of thiophene were obtained by washing the benzol with fuming sulphuric acid of sp. gr. 1·87, giving three washes of 6 per cent. of the acid in all. By this treatment the thiophene was reduced from 1·22 per cent. to 0·22 per cent. Various oxidising agents such as ozone, lead oxide, and hydrogen peroxide were tried as a means of eliminating thiophene, but while ozone gave the best results, with a low loss of benzol, the action was too slight to have any practical value. The following amounts of sulphur were found in commercial 90 per cent. benzols: From Durham tar, 250 grs.; Yorkshire tar, 240 grs.; Lancashire, 49 grs.; Staffordshire, 504 grs.; and crude benzol (coke-oven) 64·4 grs. sulphur per gallon. A. R. T.

Detection of Carbon Bisulphide in Oils. E. Milliau. (*Ann. chim. anal.*, 1912, **17**, 1-3.)—Twenty-five grms. of the oil are saponified with 10 c.c. of a concentrated aqueous solution of potassium hydroxide in the cold, the soap dissolved in about 150 c.c. of warm water, a pinch of sodium bicarbonate added, and the solution decomposed with 20 c.c. of pure hydrochloric acid. The vapours emitted are tested with lead acetate paper, which will be darkened in the case of extracted olive oil containing 0·05 to 0·1 per cent. of carbon bisulphide. No coloration is given by cotton-seed, kapok, colza, or expressed olive oils, or by extracted oils which have previously been heated for an hour at 130° C. Another test is to distil 50 grms. of the oil with 10 c.c. of pure amyl alcohol, and to collect the first 5 c.c. of the distillate. A mixture of 1 part by volume of kapok oil and 4 parts of the distillate together with a few cgrms. of powdered sulphur are now heated in a sealed tube in boiling water. In the case of expressed olive or other oil no coloration is produced even after three hours' heating, whereas an extracted olive oil containing 0·1 per cent. of carbon bisulphide gives a distinct red coloration. It is preferable to neutralise the kapok oil with sodium hydroxide before using it in the test. Cotton-seed oil may be used in place of kapok oil as a reagent, but gives a somewhat weaker coloration. The reducing agents in kapok and cotton-seed oils are not volatile with steam. No coloration is given by extracted oils after being heated for an hour at 130° C.

C. A. M.

Estimation of Cinnamic Acid by Means of Bromine in Aqueous Solution. A. W. K. de Jong.

(*Rev. trav. chim. Pays-Bas*, 1911, **30**, 223-224; through *Chem. Zentralbl.*, 1912, I., 162-163.)—When bromine is allowed to act for a long time on cinnamic acid in aqueous solution, more than 2 atoms of bromine are absorbed additively; this, however, may be avoided by operating in the following manner: The substance to be analysed, for instance, a mixture of cinnamic and benzoic acids, is dissolved in sodium hydroxide and reprecipitated with hydrochloric acid, bromine in the form of a $\frac{N}{50}$ aqueous solution is run in, and the absorption of bromine is considered to be at an end when the liquid assumes a yellow colour which is not discharged within five minutes. Potassium iodide is then added, and the iodine liberated is titrated in the usual manner.

J. F. B.

Dimethylglyoxime as a Reagent for Ferrous Salts. P. Slawik.

(*Chem. Zeit.*, 1912, **36**, 54.)—Dimethylglyoxime, which is frequently employed for the estimation of nickel, gives an intense red coloration with ferrous compounds, and may be used for their detection. One drop of a solution of a ferrous salt is mixed with a little tartaric acid and 1 c.c. of an alcoholic solution of dimethylglyoxime, and an excess of ammonia is then added. The red coloration is very intense, and the reaction exceeds in delicacy all the other tests for ferrous iron, but the colour gradually disappears as the ferrous compound becomes oxidised by the action of the air into the ferric state. This defect makes the test not very satisfactory for the detection of small traces of ferrous salts, since complete oxidation may occur owing to the rise of temperature occasioned by the addition of the ammonia. Ferric salts give the coloration on the addition of a reducing agent—*e.g.*, stannous chloride or metallic zinc.

J. F. B.

Estimation of Fat by Extraction with Cold Solvents. C. Grimme.

(*Chem. Rev. Fett- u. Harz-Ind.*, 1912, **19**, 1-5.)—Estimations made under parallel conditions with different non-inflammable solvents by the method of cold extraction were compared with those made by the ordinary method of extraction with ether. The following results, which are typical of those obtained, represent the average percentage of fat extracted from a sample of cotton-seed meal in five estimations:

Ether.	Chloroform.	Carbon Tetrachloride.	Dichlor-ethylene.	Trichlor-ethylene.	Perchlor-ethylene.	Tetrachlor-ethane.	Pentachlor-ethane.
7·45	8·58	7·43	8·13	7·46	7·79	7·71	9·62

Only in the case of carbon tetrachloride and trichlorethylene did the fat agree in amount, colour, and analytical values with that extracted with ether, the other solvents extracting substances other than fat. Further experiments showed that the following modification of Neumann's method (ANALYST, 1911, **36**, 542) was the most rapid and accurate: Ten grms. of the finely powdered sample are shaken for forty-five minutes with 100 c.c. of carbon tetrachloride, or for thirty minutes with the same quantity of trichlorethylene in a stoppered 150 c.c. flask, care being taken that the shaking is done in the direction of the vertical axis of the flask. After standing for thirty minutes, the contents are filtered (the funnel being covered with a clock glass), 50 c.c. of the filtrate evaporated, and the residue dried for an hour at 105° C., air being blown across it from time to time. The weight of fat multiplied by 20 may be taken as sufficiently accurate up to 10 per cent. Between 10 and 15 per cent. an addition of 0·2 per cent., and between 15 and 20 per cent. an addition of 0·4 per cent. should be made, these corrections being based upon the average specific gravity of fat (0·93). In comparative estimations of the amount of fat in thirty-eight samples of different seed cakes and fish meal, the results thus obtained with carbon tetrachloride and trichlorethylene agreed within − 0·16 to + 0·17 with those given by the usual method of extraction with ether. The percentages of fat extracted by the two first solvents were the same, with or without preliminary drying of the material (*Cf.* Gowing Scopes, ANALYST, 1910, **35**, 238). C. A. M.

Estimation of Mercury in Oxyphenyldimercuric Acetate and Mercurisalicylic Acid. R. Brieger. (*Arch. Pharm.*, 1912, **250**, 62-71.)—The mercury in oxyphenyldimercuric acetate may be quantitatively estimated by dissolving the salt in hot 30 per cent. acetic acid, adding an excess of standard iodine solution, and titrating the excess of iodine after twenty-four hours:

$$C_6H_3OH\ (HgOOCH_3)_2 + 4I = Hg(OOCCH_3)_2 + HgI_2 + CH_3OHI_2.$$

The concentration of the acid has an influence upon the velocity of the reaction, and by nearly neutralising the solution with potassium hydroxide the amount of mercury found was 99·2 per cent. of the theoretical amount after one hour's action. For estimating the mercury in mercuri-salicylic acid the following method is described: Half a grm. of the sample is dissolved in 30 c.c. of hot $\frac{N}{10}$ potassium hydroxide solution, and the liquid diluted to 100 c.c., and treated with a few drops of *o*-nitrophenol solution, after which 30 c.c. of $\frac{N}{10}$ hydrochloric acid are added, and the excess of acid titrated with $\frac{N}{10}$ alkali. One c.c. of the alkali consumed by the sample corresponds to 0·0138 grm. of salicylic acid, and the difference between 100 and the calculated percentage of salicylic acid, multiplied by the factor 0·5952, gives the percentage of mercury. The result thus obtained should correspond fairly closely with the value obtained as follows: A solution of 0·3 grm. of the sample in 10 c.c. of $\frac{N}{10}$ potassium hydroxide is mixed with 25 c.c. of water and 5 c.c. of 30 per cent. acetic acid, and treated 25 c.c. of $\frac{N}{10}$ iodine solution, the excess of which is titrated with thiosulphate after fifteen hours. One c.c. of the iodine solution corresponds to 0·0100 grm. of mercury, or to about 0·3 per cent. in the sample, under the prescribed method of estimation. C. A. M.

Valuation of Dynamite Glycerol. F. Hofwimmer. (*Chem. Zeit.*, 1912, **36**, 41.)—On account of the possible presence of polyglycerols the only satisfactory method for testing the value of a glycerol is a determination of the nitroglycerine obtained by nitration. The author's apparatus consists of a burette divided into 0·05 c.c., sealed at the top into a thin-walled cylindrical water-jacketed nitrating vessel, with a mark one-third from the bottom, and terminating upwards in a neck. Above it is clamped a weighed stoppered tap pipette, with two marks, to deliver 10 grms. of glycerol. This is dropped slowly into about 60 grms. of nitrating acid (45 per cent. nitric acid + 55 per cent. sulphuric acid) previously adjusted to the mark in the nitrating vessel and cooled to 15° C., while a brisk current of dried air is passed up the burette. A thermometer in the nitrating vessel is unnecessary if the cooling water is at 15-16° C., and the flow rapid. All the glycerol having been run in, the air is allowed to pass for one minute longer. After fifteen minutes' standing the acid is run out to the zero mark on the burette, the volume of nitroglycerine read off, and calculated to 10 grms. of glycerol, after weighing back the glycerol pipette. For comparison, a standard is made by similarly nitrating 10 grms. of chemically pure glycerol. The determination must always be carried out under the same conditions; the quantity of glycerol taken must be between 9·9 and 10·1 grms. The determination can be finished in an hour. O. E. M.

Alteration of Nitrocellulose and Nitrocellulose Powders by Heat. R. Fric. (*Comptes rend.*, 1912, **154**, 31.)—The effect of heat on nitrocelluloses and nitrocellulose powders is to lower the viscosity of their acetone solutions, measured by the number of seconds required for 25 c.c. of solution to flow through a tube of 2 mm. bore 20 cm. long. Thus a collodion cotton declined from 69·3 to 57·7, after heating at 80° C. for seven hours twenty-five minutes; a guncotton from 210 to 99 under similar treatment; a smokeless blank from 36 to 21 after five hours at 110° C.; and a sporting powder from 94·8 to 33·3 after twelve hours at 110° C. O. E. M.

Physical and Chemical Properties of the Petroleums of the San Joaquin Valley of California. I. C. Allen and W. A. Jacobs. (American Bureau of Mines, 1911, Bull. 19, pp. 60).—This pamphlet deals with the collection of samples and their physical and chemical examination. An electrically-heated still, suitable for difficult distillations, is figured and described, and full details are given of its mode of operation and devices employed in connection with it. Owing to trouble with frothing samples, a closed form of flash tester could not be used, and an open Pensky-Martens cup screened from air-currents was employed. Viscosity was determined in an Engler viscosimeter at 20° C. and calorific value in a Berthelot combustion bomb (Dinsmore-Atwater model). To prevent bumping a "boiling stone" of porous porcelain or pumice weighing about 0·5 grm. is found to be most useful, even with heavy oils that boil at 325° C. under a pressure of 20 mm. A large number of samples are tabulated with the analyses of their fractions. H. F. E. H.

Colorimetric Estimation of Salicylic Acid and Copper. F. Schott. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **22**, 727-728.)—Jorissen's reaction, as described by Shermann and Gross (ANALYST, 1911, **36**, 463), may be used for the colorimetric estimation of salicyclic acid and, conversely, for the estimation of small quantities of copper. In the case of salicylic acid, a definite quantity of the solution is placed in a test-tube, and in a set of exactly similar tubes are placed quantities of 0, 0·2, 0·4, 0·6, 0·8, 1·0, etc., c.c. of a salicylic acid solution which contains 100 mgrms. of the acid per litre. To each tube are now added 2 c.c. of Fehling's copper solution diluted previously with nine times its volume of water, 5 drops of a 2 per cent. potassium nitrite solution, and 5 drops of 10 per cent. acetic acid. After the volumes of the mixtures in the test-tubes have been diluted to a volume of 5 c.c., the tubes are heated in a boiling water-bath for forty-five minutes, cooled, and the coloration yielded by the solution under examination compared with those given by the quantities of salicylic acid in the other test-tubes. Salicylic acid may be estimated directly in milk by means of this method. Twenty-five c.c. of the milk are treated with 10 c.c. of Fehling's copper solution and the mixture is nearly neutralised with $\frac{N}{1}$ sodium hydroxide solution; after filtration, 15 c.c. of the filtrate are mixed with 5 drops of 10 per cent. potassium nitrite solution and the same quantity of 10 per cent. acetic acid. The coloration obtained on heating the mixture is then compared with standards prepared with milk containing known quantities of salicyclic acid. The colour of the milk serum remains unaltered in the absence of salicylic acid, but the reaction is readily obtained when the quantity of the latter amounts to 10 mgrm. or more per litre.

For the estimation of copper, comparison solutions containing 0, 0·1, 0·2, 0·4, etc., c.c. of a copper sulphate solution containing 0·3927 grm. of the crystallised salt per litre (equivalent to 100 mgrms. of copper) are employed. To each tube is added 0·3 c.c. of a 0·5 per cent. salicylic acid solution (in dilute alcohol) together with the above-mentioned qualities of nitrite and acetic acid, and the estimation is then carried out as described in the case of salicylic acid. Sucrose, dextrose, lactose, and invert sugar do not interfere with the reaction, but free mineral acids, tartaric acid, citric acid, and more than mere traces of iron should not be present in the solution under examination. The coloration is yielded by as little as 0·01 mgrm. of copper.

W. P. S.

Use of the Reagent "Tetramethyl Base." R. J. Carney. (*J. Amer. Chem. Soc.*, 1912, **34**, 32-35).—Tetramethyldiaminodi-phenylmethane $(CH_3)_4N_2(C_6H_4)_2CH_2$ in acetic acid solution was used by Trillat (*Compt. rend.*, 1903, **136**, 1205-1207) for the detection of traces of lead and manganese. The author, who finds many advantages in substituting citric acid for acetic, dissolves 2·5 grms. of the base in 10 c.c. of water containing 10 grms. of citric acid, and dilutes to 500 c.c. The base itself is prepared as follows: A mixture of 30 grms. of dimethylaniline, 10 grms. of formaldehyde, 200 c.c. of water, and 10 c.c. of sulphuric acid is warmed on the water-bath for one hour. After cooling, the mixture is made alkaline with excess of caustic soda, and the excess of dimethylaniline driven off with steam. The brownish product is cooled, filtered, and washed with water, and then recrystallised once from alcohol. The base is recommended as a new test for gold; with very dilute solutions, the reagent gives a beautiful purple colour, soon changing to blue, and then fading; the blue colour reappears on warming. The reaction is not interfered with by platinum, palladium, or other elements, and is more distinct than "purple of Cassius." Free mineral acid must be absent, so solutions should be neutralised and made slightly acid with acetic or citric acids. The test shows 0·01 mgrm. per 50 c.c. in a solution containing nothing but gold. H. F. E. H.

INORGANIC ANALYSIS.

Rapid Estimation of Carbon Monoxide. L. A. Levy. (*J. Soc. Chem. Ind.*, 1911, **30**, 1437-1439.)—The author has devised a colorimetric method for this estimation, accurate to about 0·3 per cent., which is regarded as sufficiently near for technical purposes. The method is based on that of Gautier (*Comptes rend.*, 1898, **126**, 793), in which carbon monoxide is selectively oxidised to carbon dioxide by means of heated iodine pentoxide. The author then aspirates the oxidised gas through a known volume of standard baryta solution coloured with phenolphthaleïn. The solution becomes colourless when it is completely neutralised by the carbon dioxide, the colour change thus corresponding to a fixed volume of carbon monoxide, which can be measured, and the percentage of the latter estimated. Certain precautions are necessary in carrying out the process. Unsaturated hydrocarbons are oxidised by iodine pentoxide, and must therefore be removed before the oxidation. The inhibitory action of traces of ethylene on the oxidation of carbon monoxide by the iodine pentoxide at 60° to 100° C. is not noticeable if the oxidation be effected at 170° C. The gas is bubbled through a strong solution of bromine in potassium bromide at a rate not greater than 2 litres per hour, and bromine vapour is then removed by potassium hydroxide solution (1 in 2), which also serves to remove any carbon dioxide in the original gas. After this treatment the gas is dried by passing over phosphorus pentoxide in a tube 4 inches long and 1 inch in diameter. The gas is oxidised in a U-tube containing iodine pentoxide mixed with ignited asbestos to render the column pervious to the passage of gas, and the liberated iodine is caught in a connected U-tube full of copper turnings, with which the iodine combines. This oxidation apparatus is contained in an air-oven maintained at 160° to 180° C. The oxidised gas now passes to the baryta absorption coil, a special form of which is employed in order to obtain complete absorption of carbon dioxide. The baryta solution is of such strength that it is exactly neutralised by the passage of 20 c.c. of carbon dioxide measured at 60° F. and 30 inches pressure. A three-way tap is interposed between the oxidation-tube and the baryta decolorisation vessel, so that the gas may be by-passed until the test is commenced; 100 c.c. of gas completely displaced the residual gas in the apparatus. If the volume of water flowing out of the aspirator $=\mathrm{V}$, which is the volume of gas passing, then V c.c. contain 20 c.c. of carbon dioxide, and the percentage is $\frac{2000}{\mathrm{V}}$, which can be directly read off on a specially graduated aspirator vessel. The results agree to within about 0·3 per cent. with analysis made with accurate forms of gas-analysis apparatus. The results with weaker baryta solution, in the case of gases containing only 1 to 2 per cent. of carbon dioxide, are not quite so good. Better results are obtained by using strong baryta solution with large volumes of gas, and titrating back the excess of alkali with oxalic acid. This method is useful in the analysis of fumes from stoves, mine air, and air from lime-kilns, etc. The apparatus is made in portable form by A. Wright and Co., Westminster. A. R. T.

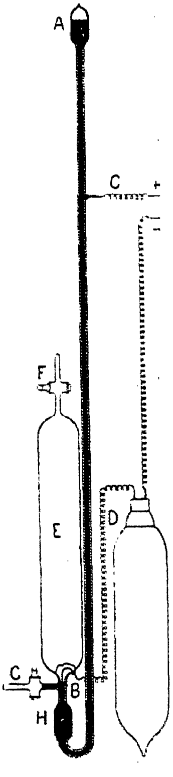
Rapid Estimation of Manganese in Ferro-Tungsten. P. Slawik. (*Chem. Zeit.*, 1912, **36**, 106.)—A mixture of 1 grm. of the finely-divided sample with 10 grms. of sodium peroxide is heated in a porcelain crucible for two or three minutes at a dull red heat, cooled, and extracted with water, acidulated cautiously with hydrochloric acid, boiled fifteen to twenty minutes with a large excess of the acid, boiled up with a slight excess of zinc oxide, and titrated with permanganate. As a check, the same sample was decomposed with sodium hydroxide and potassium nitrate in a nickel crucible, the melt dissolved in water, the manganese precipitated with sodium peroxide, filtered off, ignited, dissolved in hydrochloric acid, diluted, and, after addition of zinc oxide in slight excess, titrated hot with permanganate. Results showed a maximum error for the first method of 0·02 per cent. on an alloy containing 0·47 per cent. manganese as determined by the second. O. E. M.

Separation of Phosphomolybdates from Silicomolybdates. P. Mēlikoff. (*Comptes rend.*, 1911, **153**, 1478.)—Ammonium molybdate precipitates silicic acid as well as phosphoric acid. A mixture of equal volumes of 30 per cent. hydrogen peroxide and 8 per cent. ammonium molybdate in nitric acid dissolves out the phosphomolybdate from the precipitate, leaving the silicomolybdate. The reaction may be followed under the microscope. O. E. M.

Geochemical Interpretation of Water Analysis. C. Palmer. (U.S.A. Geological Survey, 1911, Bull. 479, pp. 31)—The interpretation of the chemical character of a water from the results of analysis is necessarily uncertain and unsatisfactory if it is based merely on the amounts of the radicles determined; a form of statement which discloses their true relations and proportions is required, and this the author, following Stabler, calls their proportional reaction capacity. The reaction capacities of the radicles of dissolved salts are the quotients obtained by dividing the weight of each radicle by its equivalent combining weight. The "reaction coefficient" is the chemical reacting power of a unit weight of a radicle, or the ratio of the reaction capacity of 1 part of that radicle to the reaction capacity of 8 parts of oxygen. Thus, calcium has an atomic weight of 40, an equivalent weight of 20, a reaction coefficient of $\frac{1}{20}$ or 0·05. The product of the "reaction coefficient" and the amount of a radicle is called the "reacting value" of that amount of the radicle, and is really equal to "mgrm. equivalents of hydrogen." When water analyses are so expressed, many operations such as the calculation of the amounts of remedial agents required to produce desired changes are much facilitated. In a particular water the reacting values of the alkali metals was 0·264, and that of the sulphates and chlorides 0·188. The excess of alkalinity (0·076) multiplied by 53 (the combining weight of sodium carbonate) is equal to 4·02, which represents the parts of sodium carbonate per million of water. It is advisable that the reacting value of a water be expressed independently of its particular concentration so as to facilitate comparison of different waters which vary in their total saline content. This is done by stating the reacting value of each radicle as a percentage of the concentration value, and so obtaining a "character formula," which not only shows the radicles found, but clearly distinguishes slight differences in the proportions of these radicles present in any waters that are to be compared. The following five properties of a water may be distinguished: (1) Primary salinity due to salts of alkalis and strong acids; (2) secondary salinity due to salts of alkaline earths and strong acids; (3) acidity or excess of strong acid if any; (4) permanent alkalinity due to alkali metals; and (5) temporary alkalinity due to alkaline earths. Carbonic acid is not regarded as capable of producing acidity, and silica and alumina are classed separately as colloids. An extensive series of analyses of American river waters is given, and the results discussed in relation to the geology of the river basins. H. F. E. H.

APPARATUS, ETC.

Thermo-Regulators. W. R. Bousfield. (*Chem. News*, 1912, **105**, 13-14.)—The figure shows an air-thermostat, capable of being used for water by slight changes of detail. This form was designed to regulate the heat of an electric radiator, worked direct from the main, carrying an alternating current at 240 volts. A, B, are the upper and lower surfaces of a mercury column in a barometer-tube, part of the column being in series with the radiator and the main current, which passes in and out of the mercury by platinum wires 1 mm. thick, sealed in the glass walls of the tube. The current enters at C, a point low enough to be always immersed in mercury. The circuit is closed by contact with the platinum wire D at B. The connected bulb E is filled with hydrogen through F, a cock, C, serving to regulate the amount of mercury for the desired temperature, and enabling E to be thoroughly washed out with hydrogen. Overheating of the thermostat expands the gas, and causes the mercury surface B to be forced away from the platinum wire, cutting off the current. The reverse process takes place when the temperature falls below the mean. The mean pressure in E must be so chosen that the mercury in A normally occupies half the bulb. The mercury in H allows of the filling of A in case of overheating, and prevents air-bubbles passing the bend at the bottom and spoiling the vacuum in A. If the mean pressure in E is about equal to atmospheric pressure, as it should be, then the tube will be of the length of the ordinary barometer-tube, and possible leakage of hydrogen through defective taps is avoided. The use of hydrogen in E is necessary, since it forms no compound with mercury, and the sparking at B does not result in oxidation nor deterioration of the surface of the mercury.



A modified form of a Lowry gas regulator is also described, which is unaffected by gas and atmospheric pressure variations, and is sensitive to 0·01° C. A. R. T.

Filtration with Alundum Plates. R. C. Benner and W. H. Ross. (*J. Amer. Chem. Soc.*, 1912, **34**, 51.)—Filtration of large volumes of liquids containing very fine precipitates by means of asbestos fibre and the Witte plate is sometimes inconvenient, and it is claimed that there is an advantage in the use of "alundum" (fused aluminium oxide) discs made by the Norton Company of Worcester, Mass., which are to be had in several grades with pores of different sizes. They fit into a glass funnel by means of an ordinary flat rubber band, and their use makes possible the stirring of the precipitate while on the plate. H. F. E. H.

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GOVERNMENT REPORT.

Milk and Cream Regulations (Draft).—The under-mentioned Draft of Regulations has been forwarded to the Society by the Local Government Board. The statutory notice of the proposal to make regulations under the Public Health (Regulations as to Food) Act, 1907, was inserted in the *London Gazette* of February 20, 1912:

To the Several Local Authorities in England and Wales for the Time being authorised to appoint an Analyst for the Purposes of the Sale of Food and Drugs Acts, 1875 *to* 1907*;—To Officers of Customs and Excise;—To the Principal Chemist of the Government Laboratories;—To Medical Officers of Health;—To Public Analysts; —To Dairymen, and to Wholesale and Retail Dealers in Milk and Cream;—and to all Others whom it may concern.*

Whereas by Section 1 of the Public Health (Regulations as to Food) Act, 1907, it is enacted as follows:

"1. The power of making regulations under the Public Health Act, 1896, and the enactments mentioned in that Act, shall include the power of making regulations authorising measures to be taken for the prevention of danger arising to public health from the importation, preparation, storage, and distribution of articles of food or drink (other than drugs or water) intended for sale for human consumption, and, without prejudice to the generality of the powers so conferred, the regulations may—

"(*a*) Provide for the examination and taking of samples of any such articles;

"(*b*) Apply, as respects any matters to be dealt with by the regulations, any provision in any Act of Parliament dealing with the like matters, with the necessary modifications and adaptations;

"(*c*) Provide for the recovery of any charges authorised to be made by the regulations for the purposes of the regulations or any services performed thereunder;

"2. For the purposes of regulations made under this Act, articles commonly used for the food or drink of man shall be deemed to be intended for sale for human consumption unless the contrary is proved. . . ."

And whereas we, the Local Government Board, are empowered by regulations made under the Public Health Act, 1896, and the enactments mentioned in that Act, to provide for the regulations being enforced and executed by the Officers of Customs and Excise, as well as by other Authorities and Officers, and subsection 2 (*a*) of Section 1 of the Public Health Act, 1896, requires that the regulations shall be subject to the consent, so far as they apply to the Officers of Customs and Excise, of the Commissioners of Customs and Excise;

And whereas in the exercise of Our powers under the Public Health (Regulations as to Food) Act, 1907, We, by the Public Health (First Series: Unsound Food) Regulations, 1908, made Regulations in relation to articles of food;

And whereas, for the prevention of danger arising to public health, it is expedient that such Regulations as are hereinafter set forth be made in relation to Milk and Cream;

And whereas the Commissioners of Customs and Excise have signified their consent to the said Regulations, so far as they apply to the Officers of Customs and Excise:

Now therefore, We, the Local Government Board, by this Our Order, and in the exercise of the powers conferred upon Us by the Public Health Act, 1875, the Public Health (London) Act, 1891, the Public Health Act, 1896, and the Public Health (Regulations as to Food) Act, 1907, and of every other power enabling Us in that behalf, do make the following Regulations, that is to say:

PART I.

ARTICLE I.—In these Regulations, unless the contrary intention appears—

(*a*) The expression "preservative substance" means any substance, preparation, or solution which when added to milk or cream is capable of retarding the onset of sourness or decomposition, or is capable of neutralising acidity (sourness) in milk or cream, and the expression "thickening substance means sucrate of lime, gelatine, starch paste, or any other substance which, when added to cream, is capable of increasing its thickness:

Provided that neither cane nor beet sugar shall be regarded as a preservative or as a thickening substance;

(*b*) The expression "milk" includes separated, skimmed, condensed, and dried milk;

(*c*) The expression "cream" means that portion of milk rich in milk fat which has risen to the surface of milk on standing and has then been removed, or which has been separated from milk by centrifugal force;

(*d*) The expression "preserved cream" means cream containing 40 per cent. or more by weight of milk fat, to which has been added—

(i.) boric acid, borax or a mixture of those preservative substances, or

(ii.) hydrogen peroxide, in amount not exceeding 0·1 per cent. by weight;

(*e*) The expression "seller" includes any person who exposes or offers for sale, or has in his possession for the purpose of sale;

(*f*) The expression "foreign" used in relation to milk, cream, or preserved cream means brought from a place situate elsewhere than in the United Kingdom, the Channel Islands, or the Isle of Man;

(*g*) The expression "Local Authority" means any local authority authorised to appoint an analyst for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907, and the expression "public analyst" means an analyst so appointed;

(*h*) The expression "Unsound Food Regulations" means the Public Health (First Series: Unsound Food) Regulations, 1908;

(*i*) Other expressions have the same meaning, *mutatis mutandis*, as in the Unsound Food Regulations.

ARTICLE II.—Subject as provided in sub-division (1) of Article V. hereof these Regulations shall come into operation on the First day of June, One thousand nine hundred and twelve.

PART II.

ARTICLE III.—(1) No person shall add, or order or permit any other person to add, any preservative substance to milk intended for sale for human consumption.

(2) No person shall sell, or expose or offer for sale, or have in his possession for the purpose of sale, any milk to which any preservative substance has been added in contravention of the provisions of sub-division (1) of this Article.

ARTICLE IV.—(1) No person shall add, or order or permit any other person to add—

(*a*) any thickening substance to cream or preserved cream;

(*b*) any preservative substance to cream containing less than 40 per cent. by weight of milk fat;

(*c*) to cream containing 40 per cent. or more by weight of milk fat any preservative substance other than—

(i.) boric acid, borax, or a mixture of those preservative substances, or

(ii.) hydrogen peroxide, in amount not exceeding 0·1 per cent. by weight;

in any case in which the cream is intended for sale for human consumption.

(2) No person shall sell, or expose or offer for sale, or have in his possession for the purpose of sale, any cream to which any thickening substance or any preservative substance has been added in contravention of the provisions of sub-division (1) of this Article.

ARTICLE V.—(1) Every seller of preserved cream shall, in every invoice, bill, advertisement, trade list, or other document which is used in connection with the sale of preserved cream for human consumption, describe that article as—

(*a*) preserved cream (boracised), or

(*b*) preserved cream (peroxidised)

as the case may require, and in no such document shall any words be contained which might imply that the preserved cream is cream to which a preservative substance has not been added.

The provision in this sub-division of this Article shall come into operation on the First day of January, One thousand nine hundred and thirteen.

(2) No seller of preserved cream shall deposit for sale in any place, or dispatch or deliver to any purchaser, or to any other person, any receptacle containing preserved cream intended for human consumption unless the receptacle is labelled in the manner prescribed in the Schedule to these Regulations, and unless, in the case of preserved cream containing boric acid, borax, or a mixture of those preservative substances, the amount of those substances calculated as boric acid (H_3BO_3) is accurately stated as not exceeding the limit specified on the label in the manner prescribed in the said Schedule:

Provided that if in any public refreshment room, restaurant, shop, or other public premises preserved cream is delivered to a purchaser or other person for consumption on the premises, the receptacle containing the preserved cream shall not be required to be labelled in accordance with the foregoing provisions if there is placed in a conspicuous position in every room on the premises in which customers are served with refreshments a notice, printed in large and easily legible type, indicating that the cream supplied for consumption on the premises is preserved cream, or if a statement to that effect is conspicuously made on every bill of fare exhibited to customers, or if by some other adequate means similar intimation is publicly given.

Article VI.—The Local Authority shall enforce and execute the Regulations in this Part of this Order, and for this purpose shall authorise the making of inquiries and the taking of any other steps which may seem to them to be necessary for securing the due observance of the Regulations in their District:

Provided that before the Local Authority institute proceedings against any person for a contravention of any of the Regulations in this Part of this Order the Local Authority shall afford him an opportunity of furnishing an explanation in writing or otherwise as they may appoint, and shall duly consider any such explanation and all the circumstances of the case.

Article VII.—Any of the officers named in Section 13 of the Sale of Food and Drugs Act, 1875, may under the direction of the Local Authority appointing him procure for examination, in accordance with the provisions of Section 14 of that Act so far as the same may be applicable, samples of milk or cream or preserved cream, or of any article resembling milk or cream, or of any substance which appears to be intended for addition to milk or cream; and the owner or occupier of any premises on which there is deposited milk or cream or preserved cream intended for sale, or for preparation for sale, for human consumption, or the person having the custody thereof, shall afford to the officer all reasonable facilities for entering the said premises for the purpose of taking samples or making inquiries or for any other purpose of the Regulations in this Part of this Order.

Part III.

Article VIII.—No person shall import into England or Wales—

(*a*) any foreign milk to which any preservative substance has been added;

(*b*) any foreign cream to which any thickening substance has been added;

(*c*) any foreign cream containing less than 40 per cent. by weight of milk fat to which any preservative substance has been added;

(*d*) any foreign cream containing 40 per cent. or more by weight of milk fat to which has been added any preservative substance other than—

(i.) boric acid, borax or a mixture of those preservative substances; or

(ii.) hydrogen peroxide in amount not exceeding 0·1 per cent. by weight;

(*e*) any foreign preserved cream which is not contained in a vessel or receptacle duly labelled in the manner prescribed in the Schedule to these Regulations.

ARTICLE IX.—(1) The Officers of Customs and Excise shall enforce and execute the Regulations in this Part of this Order and for this purpose shall, in accordance with directions given by the Treasury after consultation with Us, take samples of consignments of imported milk or cream or articles resembling milk or cream.

(2) When any Officer of Customs and Excise takes a sample in pursuance of sub-division (1) of this Article he shall divide it into not less than three parts, and send one part to the importer and one part to the Principal Chemist of the Government Laboratories, and retain one part for future comparison.

(3) In any proceeding under this Part of this Order the certificate of the Principal Chemist of the Government Laboratories of the result of the analysis shall be sufficient evidence of the facts therein stated unless the defendant require that the person who made the analysis be called as a witness, and the defendant shall not be entitled to require the person who made the analysis to be called as a witness unless he shall, at least three clear days before the return day, give notice to the prosecutor that he requires his attendance and deposit with the prosecutor a sum sufficient to cover the reasonable costs and expenses of his attendance, which costs and expenses shall be paid by the defendant in the event of his conviction.

(4) If in any case the Officer of Customs and Excise is of opinion that an offence has been committed under this Part of this Order, he shall communicate to Us for Our information the name of the importer and such other information as he possesses or may obtain as to the destination of the consignment.

PART IV.

ARTICLE X.—(1) If a difference arises in relation to any subject matter of, or to anything done under, these Regulations, the difference may, on the application of all the parties affected, be referred to Us for determination.

(2) It shall be at Our option to determine any such difference as arbitrators or otherwise; and, if We elect to determine the difference as arbitrators, the provisions of the Regulation of Railways Act, 1868, respecting arbitrations by the Board of Trade, and the enactments amending those provisions, shall apply as if they were re-enacted in these Regulations, and in terms made applicable to Us, and to the determination of the difference.

(3) Where We elect to determine any such difference otherwise than as arbitrators, We may by Our Order determine the difference, and Our determination shall be final and conclusive.

(4) Every Order made by Us in pursuance of this Article shall have effect as if the Order were enacted in these Regulations.

Article XI.—All powers given by these Regulations shall be deemed to be in addition to, and not in derogation of, any other powers conferred by Regulations under the Public Health (Regulations as to Food) Act, 1907; and those other powers may be exercised in the same manner as if these Regulations had not been made.

Article XII.—These Regulations may be cited as "The Public Health (Milk and Cream) Regulations, 1912."

SCHEDULE.

Regulations with respect to the Labelling of Preserved Cream.

1. Every receptacle containing preserved cream shall be labelled by the attachment thereto of an adhesive label, in this Schedule referred to as the "declaratory label."

2. Every declaratory label shall be in the form and contain the information indicated in Label I. or Label II. set out in this Schedule, as the case may require. Such information shall be printed in black type, of not less size than that shown in the said Label I. or Label II., upon a white ground in the centre of the declaratory label, and nothing else shall be printed on the declaratory label; except that in every case in which Label I. is used the maximum amount per cent. of boric acid (H_3BO_3) contained in the preserved cream shall be stated in figures on the declaratory label.

Label I.

PRESERVED CREAM
CONTAINING
BORIC ACID
NOT EXCEEDING
PER CENT.

Label II.

PRESERVED CREAM

PEROXIDISED

3. The size of the declaratory label shall be determined as follows:

Capacity of Receptacle.	Size of Label.		
	Inches.		Inches.
Less than ¼ pint	1	by	2⅜
¼ pint or more, but not exceeding ½ pint	2	by	3⅜
Exceeding ½ pint	3	by	5

4. The declaratory label shall be so attached to the receptacle that the whole of the label shall be visible and clearly legible, and the declaratory label shall not be overlapped by any other label or otherwise obscured.

5. The declaratory label shall not be affixed either over the mouth of the receptacle or beneath the receptacle.

6. Where on any receptacle containing preserved cream or on any wrapper or package in which such receptacle is enclosed, there appears any reference by label (other than the declaratory label) or otherwise to preserved cream contained therein, the preserved cream shall in every case be described as "preserved cream," the words "preserved" and "cream" being set out in letters of the same size.

7. No receptacle containing preserved cream shall bear or shall have attached on any label a trade description within the meaning of the Merchandise Marks Act, 1887, which shall be false or in any way likely to mislead a purchaser as to the presence or utility of the preservative substance, or as to the declaratory label, or as to the Regulations.

NOTICE.—The Public Health Act, 1896, provides by subsection (3) of Section 1 that if any person wilfully neglects or refuses to obey or carry out, or obstructs the execution of, any regulation made under any of the enactments mentioned in that Act, he shall be liable to a penalty not exceeding one hundred pounds, and, in the case of a continuing offence, to a further penalty not exceeding fifty pounds for every day during which the offence continues.

The power of making regulations under the Public Health Act, 1896, and the enactments mentioned in that Act is enlarged by the Public Health (Regulations as to Food) Act, 1907.

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REVIEWS.

THE TECHNOLOGY OF BREAD-MAKING: CHEMISTRY AND ANALYSIS OF MATERIALS USED IN BREAD-MAKING AND CONFECTIONERY. By WILLIAM JAGO and WILLIAM C. JAGO. Simpkin, Marshall, Hamilton, Kent and Co. 8vo., pp. 908. London, 1911. Price 21s. net.

The present volume is a development of the authors' former works on the same subject which appeared in 1886 and 1895, and the general mode of treatment is, in consequence, to some extent governed by the previous publications. Much space has been devoted to quite elementary considerations, such as the description of the balance and method of weighing (six pages) the principal chemical elements and their inorganic compounds, the microscope, polarisation of light, etc. The reader, whoever he may be, will probably prefer to refresh his memory on the first principles of physics and chemistry from appropriate textbooks. Without wishing to be unduly critical, we think it unfortunate that the opportunity was not taken of eliminating such unnecessary weighting from a work which is obviously intended for the practitioner rather than the student.

Mr. Jago has also devoted a considerable amount of space to recording his own researches, hitherto unpublished, on flour and fermentation. It is doubtful whether such a course is a wise one. Certainly it is not in the authors' own interests from the point of view of priority. Moreover, it is extremely annoying to investigators, as textbooks are not the recognised medium for publication of original work.

In a book of this size certain defects are unavoidable, and the reader will notice such expressions as "when a little of the substance called yeast," "sugar of milk," and "Kjeldahlise." Again, in some cases authors' names are mentioned without the accompanying reference. The account of the action of diastase on starch is only brought down to 1899, and the work of all Continental and some English workers on this subject is ignored. In consequence of this, the information relating to the dextrins of high molecular weight, which occupy an important rôle in bread-making, is quite out of date. In the list of enzymes the statement is made that invertase converts sucrose into glucose. If another edition of this book be published, the authors should carefully revise the more purely chemical and biochemical portions.

The chapter dealing with the manufacture of yeasts shows much care in its compilation, the authors having obtained some of their information first hand by visiting a well-known yeast factory in Holland. It need hardly be said that in up-to-date factories full advantage is taken of the practical bearings of Hansen's researches, and fermentation is started with pure cultures. Formulæ and full particulars are also given for the preparation of patent or bakers' home-made yeasts, Scotch flour-balms, and Parisian balms.

The important question of the strength of flour is discussed, and the work of the authors Humphries, Biffen, Wood, and Baker and Hulton is discussed at some length. Strength is defined as the measure of the capacity of the flour for producing a bold, large-volumed, well-risen loaf. In the authors' opinion there is no satisfactory method of numerically registering strength except by a baking test, when the actual volume or girth of the loaf may be measured. They consider that the strength of a flour may be deduced from the character and quality of the gluten. But this is rather begging the question, for no methods are available of expressing quantitatively the quality of a gluten. Wood has shown that certain salts exercise a marked influence on the character of gluten, and that the proteins of gluten, in the absence of electrolytes, are collectively neither ductile or tenacious; later he has stated that the determination of water-soluble phosphate gives a certain amount of information as to the character of the gluten in a flour. Wood's work, although of interest, is of a pioneer nature, and as regards the measurement of strength of flour does not carry us far. In fact, as has been pointed out by Baker and Hulton, it is not possible that the estimation of one constituent or the determination of one physical property will enable the chemist to affix a baking value to a flour, when this value is the resultant of several factors.

The authors still attach importance to gluten determinations. They consider that the weight of wet gluten is a measure of the amount of that constituent of flour which by its physical character determines the quality and nature of the resultant dough and bread.

A considerable amount of statistical information is given concerning the weights and chemical composition of typical British and foreign wheats. Different flours and milling products, and the general relationship which exists between water-absorbing power, gluten, moisture, and colour of flours, are discussed at some length.

In view of recent legislation, and reports to the Local Government Board on the bleaching of flour, the views of the authors will be read with interest. Owing to the development of milling processes, there has been a marked increase in the amount of patent flour obtainable from the wheat; but, as would be expected, this leads to a slight darkening with milling impurities. The opinion is expressed that to bleach this flour by a harmless process cannot be regarded as an adulteration, nor is such flour misbranded when called "patent flour." There will always be a difference of opinion among experts as to what is a harmless or harmful process, and in this connection it should be remembered that the United States Board of Food Inspection has decided that flour bleached with nitrogen peroxide is an adulterated product. The authors' arguments on the matter of bleaching are not convincing. The public have no means of detecting a bleached flour, and so long as bleaching is resorted to in order to improve the appearance of certain grades of flour and make it resemble a more highly priced article, misrepresentation to the advantage of the retailer is bound to ensue.

In the section on bread-making the different recipes and processes in the United Kingdom are briefly described, and also, in some detail, the diseases and faults of breads.

The nutritive value of bread is dealt with at some length, and for those who can read between the lines it will be apparent how far from disinterested was the recent Press agitation in favour of "Standard Bread."

The authors describe their methods for the estimation in flour of gluten, water-absorbing capacity, colour consistency, and other characteristics. This portion of the book will be of interest to those chemists who occasionally have to examine flours. The analysis of bread receives some attention, and the detection of various adulterations and additions.

The literature on flour and bread has been up to the present in a chaotic state, and, since the authors have made a genuine and successful attempt to present to their readers the best information available, the book should deservedly have a wide circulation. Mr. Jago has made this subject peculiarly his own, and it is not too much to say that the present volume contains the best of all that is worth knowing of flour and bread.

MODERN MICROSCOPY. By M. I. CROSS and MARTIN J. COLE. Baillière, Tindall and Cox. London. Pp. 318. Price 6s. net.

This is the fourth edition of a well-known work. In spite of its moderate dimensions, it not only deals with the design, construction and practical use of the microscope, and, to a less extent, with the principles therein involved, but also aims at furnishing a brief account of the methods of microscopical preparation employed in almost every branch of science in which the instrument is used.

The first of the three parts into which the book is divided treats of the microscope and its accessories; and the advice given as to the choice of an instrument is practical and sound, and is supported by clear statements of the reasons on which such advice is based.

The theoretical considerations underlying the construction and use of both instrument and accessories are dealt with, as far as is possible, without the use of mathematics; but the book is, and is intended to be, essentially practical, and theory is mainly referred to when necessary to give the worker an intelligent comprehension of the operations he has to undertake. Within these limits this part of the book is excellent, and to the amateur, for whom it is mainly intended, will do much to help him to understand the more elaborate treatises.

Part II. describes the various processes employed for the hardening, preserving, and embedding of animal and vegetable organisms and tissues, and the methods used for cutting, or grinding, and mounting sections of every class of material, and for making almost every kind of microscopic preparation. Coming, as it does, from the pen of Mr. Martin Cole, whose skill and experience in these matters are so well known, it is, as would be expected, thoroughly practical and efficient.

Part III. contains seven chapters, each by a separate author, on the following subjects: The Use of the Petrological Microscope; Rotifera; Fresh-Water Mites *Hydrachnidæ*); *Foraminifera*; Mosses and Liverworts; Nature Study; and, finally, the Microscopy of Foods.

This last chapter, to which many readers of this journal would naturally turn, is very unsatisfactory, and it is plain that the author of it is not familiar with the common routine work of the public analyst. In the short space at his disposal it would, of course, be quite impossible to deal adequately with such a subject; but he repeatedly goes out of his way to refer vaguely to chemical processes with which he is obviously far from conversant, and which have no connection with the microscope whatever, although to the uninitiated this might not be apparent from the text. Not only is much irrelevant matter introduced, but in very many cases the statements are incorrect or misleading. This is perhaps the more to be wondered at because the writer gives a short list of works to which readers are referred for information, and to some of which he acknowledges his indebtedness. The twenty-four photomicrographs, with which the chapter is illustrated, are in most cases extremely satisfactory; but Fig. 94 has surely been misnamed *Penicillium;* it should surely be *Aspergillus.*

Apart from this chapter, which should be entirely rewritten, the book may safely be recommended, as it deals in small space and in a thoroughly practical manner with a very wide range of microscopical technique.

C. H. Cribb.

THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

IMPORTANT NOTICE.

THE Council of the Institute of Chemistry of Great Britain and Ireland have had under consideration the particulars of proposed appointments of Public Analysts for the Metropolitan Boroughs of Lambeth and Wandsworth.

In each of these cases the following terms are set forth:

"3. The Officer will be required to examine and report upon 2,000 samples, formal and informal, or such number up to the said 2,000 as may be submitted by or on behalf of the Council in every year. For the purpose of this clause, a year shall be held to commence on the 1st January and end on the 31st December."

"4. The Officer will be required, at his own cost, and in such place as the Council may approve, to provide and maintain his own Laboratory, together with all such apparatus, chemicals, and assistance as are necessary to enable him adequately and completely to execute the duties of the office. The Officer will also be required to provide, at his own cost, and to the satisfaction of the Council and the approval of the Local Government Board, a competent Deputy to act during any absence from duty of the Officer through sickness, holiday, or other cause."

"5. In addition to the certificate of analysis directed by the Sale of Food and Drugs Acts to be given to the person submitting a sample, the Officer will be required, at the same time as he gives the Statutory certificate, to transmit direct to the Medical Officer of Health a duplicate copy of every such certificate."

"6. The fixed remuneration to be paid by the Council in respect of the said 2,000 samples, or such number up to 2,000 as may be submitted in any year, will be £500 per annum, and will be payable quarterly on the 31st March, 30th June, 30th September, and 31st December in every year."

The terms in the case of Wandsworth contain a further clause:

"7. The fee of one guinea will be paid by the Council for each analysis of water and one guinea for each attendance of the Officer at the police court when required by the Council."

The Council have also considered an announcement regarding the proposed appointment of an Analyst to the County of Antrim, under the following conditions:

"The Analyst will be paid a salary of £150 per annum, with first-class travelling expenses through the County when he is required as a witness in any case of prosecution."

"He is to make analyses of all samples submitted to him by the Inspectors appointed under the Food and Drugs Acts, and to analyse samples under the Fertilisers and Feeding Stuffs Act, 1906, for farmers and others resident in the County, at a fee of 5s. for each analysis, payable by the person sending the sample."

"Three months' notice on either side to terminate the engagement."

In explanation of the above, the Registrar has received the following letter from the Deputy Secretary to the Antrim County Council:

"In reply to yours of the 19th inst., I beg to inform you that the salary of £150 per annum to be paid to the County Analyst for County Antrim is for his services in making analyses of samples taken under the Sale of Food and Drugs Acts. In addition to this salary he is to be paid a fee of 5s. for each analysis of samples under the Fertilisers and Feeding Stuffs Act, 1906. This fee to be paid by the farmers, and other persons resident in the County, sending the samples."

"There is no limit to the number of samples likely to be submitted to the Analyst, so far as I am aware."

The number of samples per annum usually submitted is over 800. During the quarter ending September 30th, 1911, 270 samples were submitted for analysis.

The Council of the Institute are of opinion that the remuneration and conditions offered for the three appointments referred to above are not compatible with the adequate and complete execution of the duties of these offices, and that it is contrary to the public interest that appointments should be made on such terms.

The Council of the Institute view with the strongest disapproval the application for and acceptance of any of these appointments on the terms and conditions stated. They advise any Member who shall have made application for any of these appointments to withdraw the same; and they advise Members of the Institute to refrain from giving testimonials in support of any candidate applying for them.

By order of the Council of the Institute of Chemistry of Great Britain and Ireland.

RICHARD B. PILCHER,
Registrar and Secretary.

1*st March*, 1912.

The Resolution of the Council to publish this notice was reported to the Annual General Meeting of the Institute and approved.

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THE ANALYST.

OBITUARY.

We deeply regret to have to record the deaths of Mr. Arthur E. Ekins and Mr. John Pattinson, former Vice-Presidents of the Society. Obituary notices will appear in the next number of the Journal.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The Monthly Meeting of the Society was held on Wednesday evening, March 6, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous Ordinary Meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. M. E. Balston, M.A., H. A. Caulkin, B.Sc., F.I.C., and C. R. Wilkins, B.Sc., were read for the second time, and certificates in favour of Messrs. Reginald Freeman Easton, 38, Edith Road, West Kensington, W., assistant to Mr. G. Cecil Jones, F.I.C., and Leonard Goodban, A.I.C., 43, Addison Gardens, W., assistant to Mr. Frederick Davis, were read for the first time.

Miss M. Gazdar, B.Sc., A.I.C., and Mr. H. Hawley, M.Sc., were elected members of the Society.

The following papers were read: "Standards for Malt Vinegar," by A. Chaston Chapman, F.I.C.; "The Estimation of Ammonia in Carbonated Waters," by G. D. Elsdon, B.Sc., and Norman Evers, B.Sc.; "A Note on a New Preservative for Milk, Cream, etc.," by George A. Stokes; "The Phosphomolybdate Estimation of Phosphoric Acid in Soils," by S. J. M. Auld, D.Sc., Ph.D., F.I.C.; and "A Method of Estimating Calcium Carbonate in Soils," by Herbert S. Shrewsbury, F.I.C.

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NOTES ON THE DETERMINATION OF BUTTER FAT AND COCOANUT OIL IN MARGARINE.

By F. W. F. ARNAUD, F.I.C., and H. HAWLEY, M.Sc.

(*Read at the Meeting, February* 7, 1912.)

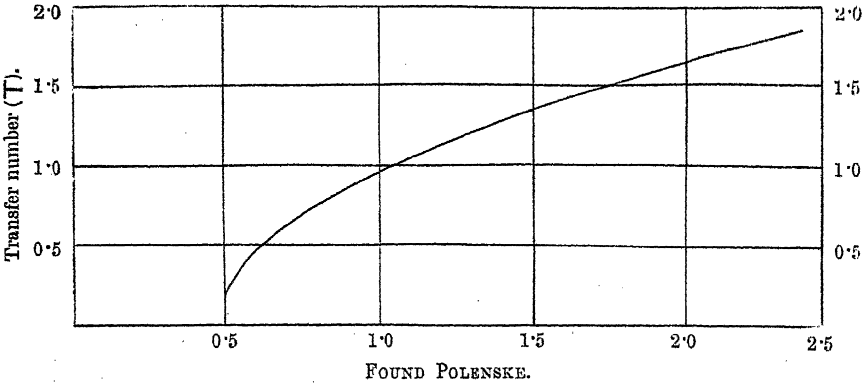
The method suggested by Cribb and Richards (Analyst, 1911, **36**, 327) for calculating from the Reichert and Polenske figures the percentage of cocoanut oil and butter fat in margarine mixtures yields excellent results with comparatively large proportions of cocoanut oil, but with quantities under 20 per cent. (the Polenske being below 2·5) the correction applied to the Reichert and Polenske figures is too great.

With the aid of the following formulæ and of the subjoined curve, deduced from the analysis of a variety of mixtures, the corrected Polenske figure may be obtained and the percentage of cocoanut oil calculated:

$$P = (P' - P'') - \frac{R}{10} + T.$$

P = corrected Polenske figure.
P′ = found Polenske figure.
P″ = Polenske figure of margarine fat free from cocoanut oil and butter (about 0·4).
T = correction ascertained from curve.

$$\text{Percentage of cocoanut oil} = \frac{100P}{17{\cdot}6}.$$



The butter fat is then found by the following method:

$$R = (R' - R'') - \text{Reichert due to cocoanut oil} - T.$$

R = corrected Reichert-Meissl figure.
R′ = found Reichert-Meissl figure.
R″ = Reichert-Meissl figure of margarine free from butter and cocoanut oil (about 0·6).
T = correction ascertained from curve.

$$\text{Percentage of butter fat} = \frac{100R}{25}.$$

The percentage of butter fat in any margarine mixture may be confirmed by the Kirschner process, for which purpose the following formula is recommended:

$$K = (K' - 0{\cdot}5) - \frac{P}{10}, \text{ in which}$$

K = corrected Kirschner figure.
K' = found Kirschner figure.
$0{\cdot}5$ = Kirschner figure of butter-free margarine.
P = found Polenske.

$$\text{Percentage of butter fat} = \frac{100\,K}{23}.$$

DISCUSSION.

Mr. C. H. CRIBB said that Mr. Richards and he had referred in their paper to the question of an allowance for the Reichert and Polenske figures of margarine fat in mixtures, but in the mixtures then dealt with there was never less than 20 per cent. of cocoanut oil and 20 per cent. of butter, so that the correction would be very small, and it seemed scarcely worth while to make it; but when the proportion of margarine might be as high as 90 per cent. it became necessary to take the correction into account.

Mr. E. R. BOLTON said that due regard must be had to the composition of the margarine. Margarine nowadays often contained notable quantities of lard, and, although lard itself had a very low Reichert-Meissl value, it exercised, when mixed with other fats, a curious altering effect on the volatile fatty acids.

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STANDARDS FOR MALT VINEGAR.

By A. CHASTON CHAPMAN, F.I.C.

(*Read at the Meeting, March* 6, 1912.)

DESPITE the numerous attempts which have been made to define malt vinegar and to lay down analytical limits for its composition, the present state of affairs in regard to that condiment can scarcely be described as satisfactory. It is clearly a much simpler matter to say what malt vinegar should be in terms of the materials used or the methods adopted in its production than to lay down chemical standards of composition. The latest pronouncement in this country appears to be contained in a letter addressed by the Assistant-Secretary of the Local Government Board to the Secretary of the London and Country Vinegar Brewers' Association. In this it is stated that malt vinegar "is derived wholly from malted barley or wholly from cereals, the starch of which has been saccharified by the diastase of malt." Under the general definition of "vinegar," moreover, it is to be "a liquid derived wholly from alcoholic and acetous fermentations; it shall contain not less than 4 grammes of acetic acid (CH_3COOH) in 100 cubic centimetres of vinegar; it shall not contain arsenic in amounts exceeding 0·0143 milligramme in 100 cubic centimetres of vinegar, nor sulphuric or other mineral acid, lead, or copper; nor shall it contain any foreign substance or colouring matter except caramel." On June 26, 1906, the United States Department of Agriculture issued a circular (No. 19) giving definitions of various kinds of vinegar. In this circular malt vinegar is defined as "a product made by the alcoholic and subsequent acetous fermentations, without distillation, of an infusion of barley malt or cereals, whose starch has been converted by malt, is dextro-rotatory, and contains, in one hundred (100) cubic centimetres (20° C.), not less than four (4) grammes of acetic acid, not less than two (2) grammes of solids, and not less than two-tenths (0·2) gramme of ash; and the water-soluble ash from one hundred (100) cubic centimetres (20° C.) of the vinegar contains not less than nine (9) milligrammes of phosphoric acid (P_2O_5), and requires not less than four (4) cubic centimetres of decinormal acid to neutralise its alkalinity."

Some time ago a certain firm of vinegar manufacturers, finding that their vinegar, although made strictly in accordance with the requirements of the United States Department of Agriculture—that is to say, wholly from barley malt and cereals, without the use of any sugar whatever—did not comply with the analytical requirements of the Department, asked me, if I could, to explain the apparent discrepancy. On referring to the above definition, it will be seen that malt vinegar must be, among other things, dextro-rotatory; and presumably any vinegar which did not comply with the definition in this respect would not be admitted into the United States under the description of "malt vinegar." Now, it so happened that, whilst the vinegar manufactured by the firm in question sometimes had a dextro-rotatory action on polarised light, it was very much more frequently appreciably lævo-rotatory. Thus, twelve samples which I examined, and which represented in the aggregate almost the whole of the vinegar at that time in stock, were all lævo-rotatory, the rotations varying from $-0·56°$ to $-0·76°$ when examined in a tube 200 mm. long, using sodium light. Inasmuch as it was absolutely certain that no sugar of any description had been used in the manufacture of this vinegar, it became necessary to explain this lævo-rotation. It was clear that certain lævo-rotatory constituents had been extracted from the grain during the manufacture of the vinegar, and a moment's consideration sufficed to show that these constituents could only be the proteins or certain of the products of their hydrolysis, such as the proteoses, peptones, and amino-acids. It may be pointed out here that the vinegar in question contained an average of 0·085 per cent. of nitrogen, equivalent to 0·53 per cent. of proteins. It is, of course, very well known that the naturally-occurring proteins are almost invariably strongly lævo-rotatory, and the same applies to many of the products of their hydrolysis, particularly the proteoses and peptones, the specific rotatory powers of these substances for sodium light varying from $-50°$ to $-80°$ in a 200 mm. tube, using sodium light. Asparagine, which occurs in malt wort, is slightly lævo-rotatory, and certain other of the amino-acids and amides are slightly dextro-rotatory. The important point, however, is that the more complex products of hydrolysis are those which are most strongly lævo-rotatory. It is clear that, if it were possible so thoroughly to ferment away the carbohydrate constituents of malt wort as to leave practically nothing undecomposed, the protein matters present might quite well cause the resulting wash to exhibit a lævo-rotatory action, and my first experiments were directed to ascertaining whether such was the case. It is, of course, the practice of vinegar manufacturers to carry out the fermentations of their worts in the presence of diastase, since by this means the malto-dextrins and other comparatively stable carbohydrates are converted into fermentable substances, and so the maximum amount of alcohol, and subsequently of acetic acid, is obtained. In the experiments described below I have therefore carried out the fermentations of the worts in question in the presence of active diastase—that is to say, the worts were not boiled prior to the introduction of the yeast. In the first place, two worts having the same sp. gr. (1070°) were prepared from the same malt, and were pitched with a small quantity of ordinary distillers' yeast, the temperature being kept within the ordinary limits of practice. The fermentations were started on December 4, and five days later the sp. gr. of each had fallen below 1000°. The following results show the optical activities in a 200 mm. tube, using sodium light, of these worts on three different dates:

	No. 1.	No. 2.
December 9	−0·40°	−0·08°
,, 11	−1·00°	−0·70°
,, 13	−1·00°	−0·80°

Two further experiments were then made with worts of rather lower sp. gr. (1050°), and employing the yeast actually used in the vinegar factory in question in place of the distillers' yeast used in the preceding experiment. The following results were obtained:

	No. 1.	No. 2.
December 11	+0·54°	—
,, 13	+0·04°	+0·20°
,, 14	−0·44°	−0·04°

It will be scarcely necessary to point out that these worts were not examined polarimetrically until their density showed that almost the whole of the carbohydrate matter had disappeared, and for that reason no considerable *plus* rotations are shown. These results show very clearly that, with a sufficiently perfect fermentation, worts prepared from malt alone are capable of yielding liquids which are appreciably lævo-rotatory.

In the next place, experiments were directed to proving that the lævo-rotation of these worts was in fact due to the presence of proteins and their products of hydrolysis. If such were the case, it should be possible, by the removal of these substances, to convert a lævo-rotatory liquid into either an inactive or a dextro-rotatory one. It is well known that, by the combined use, under proper conditions, of aqueous solutions of mercuric acetate and phosphotungstic acid, both the proteins and the majority of the products of their hydrolysis may be removed from organic liquids without effecting any change in the carbohydrates present. The mercuric acetate solution used in the following experiments consisted of a saturated aqueous solution of the salt; the phosphotungstic acid solution consisted of a 25 per cent. solution of the substance in water without the addition of mineral acid. In the first place, the mercuric acetate and the phosphotungstic solutions were added to some fermented malt wort made in the laboratory, and having a rotation in a 200 mm. tube of −0·45°. After filtering and making the requisite correction for the dilution due to the volume of the reagents employed, the liquid was found to have a rotation of +0·46°. Similar experiments were then made with five samples of lævo-rotatory vinegar, with the following results, the numbers under (*a*) representing the rotation of the vinegar prior to the addition of the reagents, and those under (*b*) the rotation after filtration:

	(*a*)	(*b*)
No. 1	−0·63°	+0·02°
„ 2	−0·60°	inactive
„ 3	−0·65°	+0·08°
„ 4	−0·63°	+0·13°
„ 5	−0·72°	+0·06°

These results entirely bear out those obtained with the malt wort itself, and show that the lævo-rotation of this vinegar is, in fact, due to the presence of proteins and the products of their hydrolysis. During the process of fermentation the amino-acids, and to a less extent the proteoses and peptones, will have been utilised by the yeast for its nutrition, and the residual nitrogen in the vinegar must consist largely of proteins and of their more complex hydrolytic products—that is to say, of those substances which exhibit the greatest degree of lævo-rotation. I may perhaps add that I have made experiments with the mercuric acetate and phosphotungstic acid solutions in the presence of a small quantity of lævulose, and have found that when used under proper conditions these reagents bring about no change whatever in the rotation of that carbohydrate. This is borne out by experiments which have recently been made by Neuberg and Ishida (*Biochem. Zeitsch.*, 1911, **37**, 142-169), who call attention to the errors which may result in the polarimetric estimation of sugar in natural products owing to the presence of lævo-rotatory proteins and amino-acids. These authors have also made use of mercuric acetate and phosphotungstic acid, and have recorded a great many results to show that these reagents have no effect on the rotation of the carbohydrates present. The above results show very clearly that a genuine malt vinegar—that is to say, a vinegar prepared entirely from malt and cereals whose starch has been converted by diastase—need not necessarily be dextro-rotatory, but may be either lævo-rotatory or optically inactive. As further showing the fallacy of insisting on dextro-rotation as an indication of genuineness, it is only necessary to point out that the unfermentable matter in many samples of commercial invert sugar is optically inactive, and that even if a considerable proportion of invert sugar were used in the manufacture of the vinegar, the latter would not necessarily be lævo-rotatory. It would appear, therefore, that unless the United States Department of Agriculture modify their existing regulation, considerable injustice may be done, not only to the home producer, but also to vinegar manufacturers in other countries, whose malt vinegar may be optically inactive or slightly lævo-rotatory, and who may wish to introduce it into the United States. Whilst many samples of malt vinegar are undoubtedly dextro-rotatory, to insist upon this analytical requirement would be tantamount to excluding from the United States the vinegar of every manufacturer who is successful in very thoroughly fermenting away the carbohydrate constituents of his worts, and would practically amount to putting a premium on bad working, to say nothing of tempting dishonest manufacturers to make their vinegar comply with the requirements by the appropriate additions. Perhaps the simplest way of manufacturing a malt vinegar to insure its satisfying the United States authorities in respect of its rotation would be to employ in its manufacture a proportion of commercial glucose.

The requirements of the United States Department of Agriculture in respect of the proportion of soluble phosphoric acid in the ash of malt vinegar affords a further example of the inadvisability of putting forward standards for manufactured products, the composition of which must in the nature of things depend somewhat considerably on the precise methods adopted in their manufacture. The amount of phosphoric acid passing from the grain into the wort, as also that left in the soluble condition when the resulting vinegar is incinerated, must depend very largely on the mineral composition of the water used for mashing. Although this is sufficiently obvious, it may perhaps be of interest to record the following experiments which illustrate this point. Two different malts were taken, and were mashed (*a*) with distilled water, (*b*) with water containing about 20 grains per gallon of total dissolved solid matter, and (*c*) with a very hard water containing more than 100 grains per gallon of calcium sulphate. The mashes were made under precisely the same conditions of time and temperature, and were, in fact, treated in the same way throughout. At the end of the mashing period of one and a quarter hours the mashes were filtered, and the total phosphoric acid determined in the worts in the usual way.

MALT A.	Phosphoric Acid (P_2O_5).
Distilled water	46·56 grains per gallon.
Water containing 20 grains of total dissolved solid matter ...	42·44 ,, ,,
Very hard water	30·44 ,, ,,

MALT B.	Phosphoric Acid (P_2O_5).
Distilled water	44·77 grains per gallon.
Water containing 20 grains of total dissolved solid matter ...	37·61 ,, ,,
Very hard water	26·88 ,, ,,

I may add that in the preparation of the above worts the same proportions of malt and water were used in all cases. I have at different times examined several samples of genuine malt vinegar made with very hard water, and have found that when ignited they yielded ashes which contained almost the whole of the phosphoric acid in the insoluble condition—that is to say, as tribasic calcium phosphate. On extracting these ashes with water, the resulting solutions were practically free from phosphoric acid, and were almost neutral. I think I have shown that the United States definition of malt vinegar requires amendment in at least two respects, and have incidentally provided yet another example of the dangers of setting up official standards for the composition of manufactured foodstuffs.

My best thanks are due to my assistant, Mr. R. L. Collett, for help in connection with this investigation.

A METHOD OF ESTIMATING CALCIUM CARBONATE IN SOILS.

By HERBERT S. SHREWSBURY, F.I.C.

(*Read at the Meeting, March* 6, 1912.)

Methods of estimating calcium carbonate in soils depending on the measurement of carbon dioxide evolved from the carbonates by absorption or otherwise have not been found very satisfactory; the modified method proposed by F. S. Marr (*J. Agricultural Science*, 1909, **3**, 155) appears to give good results, but is somewhat lengthy, and entails rather special apparatus and skilful manipulation for its successful application.

An alternative method which I have devised depends upon the extraction of the calcium carbonate from the soil in $\frac{N}{4}$ acetic acid, the conversion of the calcium acetate so formed into calcium oxide and its solution in $\frac{N}{10}$ acetic acid, and the titration of the excess of acid required by the lime with $\frac{N}{10}$ potash or soda, using phenolphthaleïn as an indicator.

Ten grms. of finely divided air-dried soil are placed in a dry wide-mouthed flask or bottle, and 100 c.c. of approximately $\frac{N}{4}$ acetic acid are added. For a period of ten minutes the flask is shaken with a rotary movement every time the bulk of the soil settles, which occurs about every thirty seconds. The soil is then allowed to settle for the last time, and the liquid contents of the flask decanted through a dry filter. Schleicher and Schüll's filter cones answer the purpose admirably. A control experiment is made with another 10 grms. of soil, using distilled water as the extracting solvent. Twenty-five c.c. of each filtrate are evaporated to dryness in a platinum dish, and ignited at a bright red heat for thirty minutes, the residues when cool being treated with 10 c.c. of $\frac{N}{10}$ acetic acid (or more if necessary). Solution is effected in a few minutes, particularly if the residues are gently rubbed with a glass rod tipped with a small rubber bung.

The excess of acid is then titrated with $\frac{N}{10}$ soda or potash, using phenolphthaleïn as indicator. The volume obtained from the control is now subtracted from that obtained from the acid extraction of the soil, and the result multiplied by 0·2. The product gives the percentage of calcium carbonate in the soil.

Notes on the Process.—The quantities given will answer for soils containing up to 5 per cent. of calcium carbonate, which covers the usual range. For larger percentages proportionate amounts of soil are taken. The time and method of extraction were based on experiments made with pure calcium carbonate and $\frac{N}{4}$ acetic acid.

Some soils appear to retain a portion of the calcium acetate, presumably by reason of a greater concentration of the solution occurring in immediate contact with the soil particles. The occasional error due to a soil of this type does not appear to be large enough to warrant a complete washing of the soil, an operation which is lengthy and tedious.

The purpose of the control is to measure alkaline carbonates and alkaline organic salts, soluble organic calcium salts, calcium nitrate, or any bodies which are soluble in water, and which on ignition leave an alkaline residue soluble in $\frac{N}{10}$ acetic acid. One-tenth c.c. of $\frac{N}{10}$ acid was the maximum obtained in this titration for three soils that were examined. $\frac{N}{10}$ acetic acid is chosen for the solution of the residues because it does not dissolve ignited iron oxide, whilst easily attacking calcium oxide.

It is preferable to measure the calcium oxide by titration rather than to weigh it or estimate it by conversion into oxalates and comparison of the turbid liquid with standards, since the first method excludes other salts of calcium than the carbonate, with the possible exception of organic calcium salts insoluble in water but soluble in $\frac{N}{4}$ acetic acid. It is desirable to make qualitative tests on the same volumes of the acetic acid extract of the soil for the presence of calcium and magnesium, chlorides and sulphates.

Frequently the last three are only present in traces.

If the amount of magnesium is comparatively large, it may be quickly estimated by the method given by Thresh ("Examination of Water and Water-Supplies," p. 238), depending on the comparison of the precipitate obtained with sodium hydrogen phosphate (after removal of calcium with ammonium hydrate and ammonium oxalate), when shaken up in a certain volume of water, with standards prepared in the same way. The magnesium estimated would of course include any salt of magnesium soluble in $\frac{N}{4}$ acetic acid, but in most soils the error introduced would be insignificant.

The following table sets out the results of test experiments, made with the process on soils prepared with known percentages of calcium carbonate. The sample of precipitated chalk used for this purpose was analysed, and found to contain practically 100 per cent. of calcium carbonate:

Sample of Soil.	Per Cent. Calcium Carbonate in Soil.			
	Originally Present.	Added.	Total.	Found by Process.
A. Surface soil from soft ferruginous shale ...	0·07	0·05	0·12	0·13
B. Heavy clay subsoil previously treated with hot concentrated hydrochloric acid for twelve hours, washed, and dried in steam-oven	Nil	0·50	0·50	0·50
C. Clay subsoil	0·29	0·50	0·79	0·61
D. Clay subsoil	0·23	4·76	4·99	4·86

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THE PHOSPHOMOLYBDATE ESTIMATION OF PHOSPHORIC ACID IN SOILS.

By S. J. M. AULD, D.Sc. (Lond.), Ph.D., F.I.C.

(*Read at the Meeting, March* 6, 1912.)

The necessity of estimating very small quantities of phosphoric acid during the chemical analysis of soils has lead to the suggestion of many methods for the direct utilisation of the bulky phosphomolybdate precipitate, instead of finally precipitating its ammoniacal solution with magnesia mixture.

The difficulties attending these methods are concerned chiefly with the variation in composition of the yellow ammonium phosphomolybdate precipitate, the constitution of which has been the subject of much investigation. Under standard methods of precipitation, however, any variation in composition is practically confined to the amount of ammonia in combination and to the quantity of water of crystallisation; this, of course, apart from the possible co-precipitation of molybdic acid, admixture with other ammonium salts or incomplete separation (in soil analysis) from silica, etc. The ratio of $P_2O_5 : MoO_3$ was originally stated by Debray and others to be 1 : 20, but was definitely shown by Finkener (*Ber.*, 1878, **11**, 1638) and Gibbs (*Amer. Chem. J.*, 1882, **3**, 317) to be $P_2O_5 : MoO_3 = 1 : 24$. This has been confirmed more recently by Levi and Spelta (*Gazz. Chim. Ital.*, 1903, **33**, 207). Gibbs gives the formula of the yellow precipitate as

$$(NH_4)_3PO_4{\cdot}12MoO_3 + (NH_4)_2HPO_4{\cdot}12MoO_3 + 8H_2O;$$

whilst Finkener describes the precipitate, washed with ammonium nitrate solution and carefully heated in a crucible until the ammonium nitrate is volatilised, as

$$(9 - x)(NH_4)_2O{\cdot}xH_2O{\cdot}3P_2O_5{\cdot}72MoO_3,$$

where x generally equals 1. Carnot (*Bull. Soc. Chim.* 1893, **9**, 343) regards the reprecipitated compound as

$$3(NH_4)_2O{\cdot}P_2O_5{\cdot}24MoO_3{\cdot}3H_2O,$$

and Hundeshagen (*Zeit. f. Anal. Chem.*, 1889, **28**, 141) states that the precipitate dried at 130° C. is always

$$(NH_4)_3PO_4{\cdot}12MoO_3,$$

independent of the content of mineral acids, phosphoric and molybdic acids, or ammonium salts. The ratio $NH_4 : P_2O_5$ is thus variously given as 5 : 1, 16 : 3, and 6 : 1. This may possibly be due to the fact, shown by Levi and Spelta (*loc. cit.*) that phosphomolybdic acid contains twenty-seven replaceable hydrogen atoms, five of which behave differently from the remaining twenty-two. The estimation of phosphoric acid by Carnot's method (*loc. cit.*), by weighing the reprecipitated compound after drying at 100° C., which is still extensively used and recommended in certain textbooks, should therefore be rejected on the grounds of insufficient evidence of composition; besides which, the coefficient recommended by him—viz., 0·0373—is calculated on the *fully hydrated* precipitate of the formula given above. On the other hand, the factor 0·043 recommended by the Committee of the Association of Official Agricultural Chemists of the United States has been recognised as inaccurate (*cf.* Wiley's "Agricultural Analysis, i. 423). To test the possible removal of water of hydration during drying, and the composition of the residual precipitate, an extended series of analyses by the Carnot method were carried out, using standard solutions of purified sodium phosphate to check the results, which were also controlled by dissolving the precipitates in ammonia solution and precipitating with magnesia mixture. Previous to drying, the precipitates were thoroughly washed with a 1 per cent solution of nitric acid.

TABLE I.

Series.	Volume of $\frac{N}{5}$ Na_2HPO_4 Solution.	Weight of P_2O_5.	Precipitate dried at 100° C.		Precipitate dried at 105° to 110° C.	
			Average Weight.	Factor.	Average Weight.	Factor.
	C.C.	Grms.	Grms.		Grms.	
1	20	0·0946	2·5165	0·0376	2·5045	0·0378
2	15	0·0710	1·8761	0·0378	1·8732	0·0381
3	10	0·0473	1·2671	0·0375	1·2443	0·0381
4	5	0·0236	0·6300	0·0376	0·6185	0·0383

Although carried out with care, a number of the estimations gave only approximate results, and these were not included in the above table. The method was certainly not completely satisfactory, and if used should probably be calculated from the coefficient 0·0378, the theoretical value for the anhydrous salt. This, however, probably does not represent the true state of affairs, and it will be noticed that heating for some time at 105° to 110° C. gives a residue containing a percentage of phosphoric acid incompatible with Hundeshagen's asserted stability of the ammonium salt, assuming that his simple formula correctly represents its composition. This tends to confirm the statement of Dehérain ("Chim. Agricole," p. 449) that decomposition may ensue if the yellow precipitate is heated above 90° C. He recommends the factor 0·0376.

It was chiefly, however, for the purpose of examining the more generally adopted procedure of gravimetrically estimating the P_2O_5 in soils that this work was started. The operation, first suggested by Hehner (ANALYST, 1879, 4, 23), consists of dissolving the ammonium phosphomolybdate precipitate in dilute ammonia, evaporating the ammoniacal solution, and weighing the residue after drying or gentle ignition. The factors employed for calculating the percentage of phosphoric acid vary somewhat according to different authors and works of reference, but, from their order of magnitude, apparently frequently depend on an assumed stability of the combined ammonia on heating at moderate temperatures:

Mode of Treatment.	Factor.	Reference.
Heat gently to volatilise the ammonium nitrate	0·0374	Finkener (*loc. cit.*).
Ignite gently over an Argand burner	0·0379	Hall ("The Soil," p. 146).
Repeated evaporation to dryness with small quantities of water ...	0·0350	Hehner (*loc. cit.*, p. 27).
Heat to dark blue colour	0·0396	Wood ("Pract. Agric. Chem.," p. 17).

The difference in the factors becomes of importance when one is forced to estimate very small quantities of P_2O_5, as, for example, in the estimation of available or "citric soluble" phosphoric acid in soil. In this case there is difficulty in increasing the amount of material used, and the weight of the final residue may not amount to more than 0·3 to 0·4 grm. During the heating, the residues generally take on a more or less blue colour—sometimes to a very considerable extent—and this cannot be controlled to any particular stage coincident with the complete removal of the extraneous ammonium salts. Hehner avoided this by washing the precipitate with dilute alcohol, dissolving in ammonia, and weighing the residue after repeated evaporation to dryness with small quantities of water.

The formation of the blue colour generally obtained would also indicate a partial decomposition of the phosphomolybdate, and suggest *prima facie* that the residue should be obtained either wholly yellow or wholly blue. That one is not so likely to obtain a uniform product at all by dissolution of the yellow precipitate in ammonia may be gathered from a consideration of the reactions involved.

According to Levi and Spelta (*loc. cit.*), ammonia resolves ammonium phosphomolybdate into ammonium phosphate and ammonium molybdate. Certainly, repeated evaporation of the ammoniacal solution with water gives acid molybdate and ammonium phosphate. But the reaction is probably not quite so simple as might be expressed by the equation

$$x(NH_4)_2O{\cdot}P_2O_5{\cdot}24MoO_3 + 2(27-x)NH_4OH = 24(NH_4)_2O{\cdot}MoO_3 + \\ 2(NH_4)_3PO_4 + (27-x)H_2O,$$

and may, at any rate partially, involve the formation of ammonium phosphopentamolybdate:

$$x(NH_4)_2O{\cdot}P_2O_5{\cdot}24MoO_3 + 2(22-x)NH_4OH = 19(NH_4)_2O{\cdot}MoO_3 + \\ 3(NH_4)_2O{\cdot}P_2O_5{\cdot}5MoO_3 + (22-x)H_2O.$$

This would serve as an explanation of the separation from the ammonia solution by Gibbs (*loc. cit.*), under certain conditions, of Debray's duodecaphosphomolybdate, since the pentaphosphomolybdate salts in certain circumstances split off phosphoric acid:

$$4(5MoO_3{\cdot}P_2O_5) + 9H_2O = 20MoO_3{\cdot}P_2O_5 + 6H_3PO_4.$$

It is difficult to see, therefore, how a satisfactory product for weighing can be obtained by heating the residue at 100° C. or by partial ignition, since ammonium phosphate readily loses ammonia on heating, and the molybdate is partially reduced with the formation of blue molybdic oxide (Mo$_3$O$_8$). There would also appear a possible danger, by overheating, of loss of phosphoric acid. By the ordinary method used in the analytical laboratories of the South-Eastern Agricultural College, the residue is heated over an Argand burner for some time, and weighed in a partially blue state. Taking at random the residue in five phosphoric acid estimations in soils, and further heating, till uniformly blue, over a medium-sized Bunsen flame, different percentage losses were obtained, although the blue residues after moderately strong heating for another half-hour in no case lost any further weight.

TABLE II.

Soil.	Weight of Residue; partially Blue.	Weight of Residue; wholly Blue.	Loss on Heating.
	Grms.	Grms.	Per Cent.
1	0·0770	0·0710	7·5
2	0·1595	0·1535	3·7
3	0·0981	0·0909	7·1
4	0·1452	0·1390	4·1
5	0·0679	0·0650	4·4

The effect of heating until uniformly blue and of constant weight was consequently tried with precipitates dissolved in ammonia obtained from standard phosphate solutions, and also with soil extracts, in order to determine the constancy of the P$_2$O$_5$ content. The results are given in Tables III. and IV.

TABLE III.

Solutions of $\frac{N}{5}$ Sodium Phosphate.

Weight of P$_2$O$_5$ taken.	Partially Blue Residue.		Loss on Ignition.	Blue Residue.	
	Weight.	Factor.		Weight.	Factor.
Grms.	Grms.		Per Cent.	Grms.	
0·0946	—	—	—	2·3995	0·0394
0·0473	1·2800	0·0372	4·6	1·2215	0·0388
0·0460	1·2153	0·0379	—	—	—
0·0236	0·6650	0·0356	3·7	0·6111	0·0387
0·0710	1·9271	0·0368	4·2	1·8450	0·0385
0·0710	1·9280	0·0368	5·5	1·8221	0·0389
0·0946	2·5002	0·0378	4·1	2·3969	0·0394
0·0946	2·5256	0·0375	4·8	2·4050	0·0393

TABLE IV.

Soil Extracts.

Soil.	P_2O_5 Content of Extract.	Partially Blue Residue.		Loss on Ignition.	Blue Residue.	
		Weight.	Factor.		Weight.	Factor.
	Grms.	Grms.			Grms.	
Q.C. ...	0·0057	0·1501	0·0383	2·7	0·1460	0·0396
P. 1 ...	0·0064	0·1626	0·0393	1·4	0·1620	0·0399
P. 2 ...	0·0063	0·1640	0·0389	2·4	0·1600	0·0399
Q.B. ...	0·0064	0·1651	0·0392	3·0	0·1601	0·0401
H. ...	0·0071	0·1823	0·0389	2·3	0·1781	0·0398

This series of soils was simply one of many tested during the ordinary routine analytical work, and the residues in Column 3 give the actual weights taken for calculation. The difficulty of testing the soil content sufficiently accurately by estimation as magnesium pyrophosphate may be responsible for the high factor obtained, but the experience gained in these numerous analyses rather points to a difficulty of preventing the decomposition of the yellow compound where the amount is small, and the gradual attainment of a more uniform weight and factor on heating. From this point of view the figures in Tables III. and IV. are very suggestive, the rather bluer "yellow" residues from the smaller phosphoric acid content of the soils giving higher, but more concordant, factors than those from the yellower residues of Table III. It must be remembered, also, that the residues in Column 3 of Table IV. were obtained by Mr. R. H. Carter, the College analyst, in the ordinary course of practice, and in the case of the higher factors for P. 1 and Q.B. the residues were much more blue than the others.

The experiments would therefore seem to show that residues containing, on the average, 3·891 per cent. of phosphoric acid are generally obtained by the method described by Hall, as against the 3·794 actually laid down. The factors obtained by heating the ammoniacal solution residues to complete blueness are more concordant than those obtained in practice from the yellow residues, and average 0·0393; but, in harmony with the non-uniformity of the residue, as shown above, the results vary somewhat.

Much better results were obtained by heating the yellow ammonium phosphomolybdate precipitate direct until it is of a blue colour. This method is described by Wood (*loc. cit.*). He gives the blue residue the same formula—viz., $P_2O_5 \cdot 24MoO_3$—as that obtained by heating the residue after dissolution in ammonia, and uses the same coefficient for calculating the results—viz., 0·0396.

Owing to the necessity of freeing the yellow precipitate from silica or other contamination, the procedure described below was eventually adopted.

Method Recommended.—The yellow precipitate obtained in the ordinary way is dissolved in dilute ammonia, and the filtered solution reprecipitated by the addition of excess of nitric acid (1 part of acid to 2 parts of water). A further 5 c.c. of ammonium molybdate solution are also added, and the mixture is allowed to stand in a warm place for some time, as in the first precipitation. The precipitate thus obtained is of a fine consistency, and may be completely filtered with ease and rapidity through a Gooch crucible. It is then washed thoroughly with 1 per cent. nitric acid, dried in the oven, and ignited until of a uniform dark blue colour and of constant weight. During ignition the base of the Gooch should be protected by a cap or by placing it inside another crucible.

A quicker method, useful for students, and nearly as accurate, consists of reprecipitating the ammonia solution with nitric acid in a porcelain dish, washing out most of the ammonium nitrate by decantation, and, after drying on the water-bath, igniting until uniformly blue. Very little phosphoric acid remains in solution after addition of the nitric acid.

The results obtained by the method are given below:

TABLE V.

Experiment.	Weight of P_2O_5 taken.	"Yellow" Residue.		Blue Residue.	
		Weight.	Factor.	Weight.	Factor.
	Grms.	Grms.		Grms.	
1	0·0473	1·2391	0·0382	1·2015	0·0395
2	0·0473	—	—	1·2010	0·0395
3	0·0236	—	—	0·5970	0·0396
4	0·0236	0·6250	0·0379	0·5975	0·0396
5	0·0710	—	—	1·7925	0·0396
6	0·0946	2·4705	0·0383	2·3800	0·0397
7	0·0236	0·6255	0·0380	0·5961	0·0396
8	0·0236	0·6230	0·0379	0·6011	0·0394

The factors from the yellow resides vary much as before, but by careful manipulation very constant results were obtained with the blue residues, the average being 0·0396.

It is interesting to find that the coefficient obtained does not correspond to the phosphomolybdic acid ($P_2O_5 \cdot 24MoO_3$), which contains 3·94 per cent. of P_2O_5, but to a partial reduction corresponding to the formula $P_2O_5 \cdot Mo_3O_8 \cdot 21MoO_3$.

SUMMARY OF RESULTS.

1. Owing to the difficulty of controlling the amount of ammonium salts present, and the possible variation of the amount of combined ammonia, methods of weighing the phosphomolybdate precipitate direct should be avoided. If used, Carnot's method should be employed, using the factor 0·0378.

2. If the precipitate is dissolved in ammonia, the difficulty of removing ammonium salts without, in practice, decomposing the residue to an indefinite extent renders it desirable to weigh the residue in the uniformly blue condition. In

the ordinary method of heating till partially blue, the factor used should be 0·0389; if heated until quite blue, 0·0393.

3. Since ammonia splits up the ordinary phosphomolybdate precipitate on solution, and the P_2O_5 content of the residue may be subject to variation, it is best to reprecipitate the ammoniacal solution with nitric acid, filter through a Gooch crucible, wash, and ignite the residue until dark blue in colour and of constant weight. Factor = 0·0396.

4. As a quicker method of carrying out the operation, the reprecipitated phosphomolybdate may be washed by decantation, evaporated to dryness on the water-bath, and the residue ignited direct, using the same coefficient for calculating the result.

The author wishes to thank Mr. F. Knowles for carrying out some of the analyses quoted above.

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DISCUSSION.

Dr. DYER said that ever since Hehner read his paper in 1879, he had adopted Hehner's modification of the molybdic acid process for the determination of such small quantities of phosphoric acid as were contained in soil. If, after the ammoniacal solution was evaporated down, the residue was again evaporated two or three times with water, as Hehner had suggested, a precipitate was obtained which could be rapidly dried in the water-oven to a constant weight. If the evaporation with water were omitted, the residue had to be kept in the water-oven for a much longer time.

The PRESIDENT said that he used the molybdic acid method for the determination of phosphorus, chiefly in the case of steel and iron. The composition of the compound was perfectly definite, provided it was precipitated under definite conditions, which conditions, of course, must be duly learnt. Having obtained the precipitate under those definite conditions, he thought it immaterial by what method it was weighed. He had never found any difficulty in obtaining correct results by drying the precipitate in a porcelain crucible at 100° C. in the water-oven, the precipitate being rinsed from the filter-paper into the crucible and dried and weighed. Latterly, however, he had adopted the more rapid method of Handy—namely, dissolving the precipitate from the filter with a standard solution of caustic soda and titrating back the excess of soda with standard nitric acid solution. When it was desired to check the results, a larger quantity of steel was taken, and the larger molybdate precipitate was redissolved, and the phosphorus precipitated with magnesia mixture and weighed as magnesium pyrophosphate. The results commonly agreed within a point or two in the third decimal place, which showed that the composition of the molybdate precipitate was quite definite, and also that its reaction with caustic soda was definite, so that probably its reaction with ammonia would be equally definite.

Dr. AULD, in reply, said that the estimation of phosphoric acid in soil called for a nearer approach to absolute accuracy than many other chemical estimations, because in the conversion of the results, say, into pounds per acre, any error that might exist was very largely multiplied. He therefore could not agree that it was not desirable to get as near to scientific exactitude as possible. It was only recently that his attention had been directed to Hehner's method, and he had never seen it used—perhaps because the washing of the precipitate with alcohol and its repeated evaporation with water were regarded as troublesome—and from that point of view it was an advantage to be able to weigh the precipitate at once without further manipulation. He agreed that the composition of the precipitate was, practically speaking, constant, but with very small quantities* the error due to the use of an inexact factor became relatively large. He had found titration to give good results, but in order to keep the experimental error of reading within proper limits about 12 c.c. of liquid must be used in an ordinary burette, and with soils the precipitate was usually too small to allow of this unless the quantity worked upon was inconveniently large.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Micro-Chemical Detection of Certain Alkaloids. A. Grutterink. (*Zeitsch. anal. Chem.*, 1912, **51**, 175-234.)—The reactions between alkaloids and many other substances are described, and particular information is recorded as to the crystalline compounds which are formed as the result of these reactions. In many cases the crystals are characteristic, and may be obtained from very small quantities of the alkaloids. In the original paper the reactions are recorded in tabular form, and those relating to the alkaloids of the strychnine, cocaine, quinine, and calumba groups receive special attention. Many of the natural and synthetic bases may be identified microscopically, the following organic acids yielding characteristic compounds or crystals: *m*-nitrobenzoic acid (with strychnine); *p*-nitrobenzoic acid (with strychnine and tropacocaine); dinitrobenzoic acid (with hydrastine, novocaine, brucine, and strychnine); trinitrobenzoic acid (with novocaine, tropacocaine, strychnine, brucine, and conine); dinitro-anisic acid (with hydrastamide); dihydroxybenzoic acid (with cinchonine); trihydroxybenzoic acid (with quinidine); opianic acid (with brucine); meconic acid (with quinidine); mellitic acid (with quinidine and cinchonidine); naphthalenesulphonic acid (with cocaine and strychnine); *p*-nitrophenylpropiolic acid (with hydrastinine, hydrastine, strychnine, tropacocaine, cinchonidine, nicotine, and hydrastamide). Potassium permanganate is also a very useful reagent for the micro-chemical detection of hydrastinine, hydrastine, tropacocaine, and cotarnine. W. P. S.

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* Such as were obtained in estimating the "citric soluble" phosphoric acid in soil, and in which the weight of residue might not be more than 0·03 grm.

Process of Extracting Alkaloids from Syrups. E. Kohn-Abrest. (*Bull. Soc. Chim.*, 1912, **11**, 73-75.)—When a syrup containing alkaloids is treated with alcohol and solid potassium carbonate, the alcohol, in causing the dehydration of the potassium salt, simultaneously precipitates the sugar, whilst the alcohol forms a supernatant layer containing the whole of the alkaloids present. Syrups of sp. gr. 1·15 to 1·20 should be shaken with about four times their volume of absolute alcohol and about their own weight of potassium carbonate, the shaking being repeated several times, and the mixture then left for twelve hours. In the case of more concentrated syrups it is preferable to use 95 per cent. alcohol to insure complete elimination of sugar from the alcoholic layer. After standing for a sufficient time, the alcoholic extract is decanted, filtered, and distilled, and the residue taken up with a little absolute alcohol and again filtered. The fresh residue is dissolved in 10 to 15 c.c. of boiling chloroform, the filtered solution evaporated, the residue dissolved in hydrochloric acid, the solution evaporated to dryness, and the hydrochloride of the alkaloid crystallised. The method has given excellent results with syrups containing morphine, codeine, quinine, strychnine, and atropine. Thus, 20 grms. of a syrup containing 0·040 grm. of morphine hydrochloride gave a residue of 0·15 grm., which, after purification as described, yielded 0·040 grm. of crystals consisting entirely of morphine hydrochloride. C. A. M.

Estimation of Glycyrrhizic Acid in Liquorice Juice. H. Cormimbœuf. (*Ann. Chim. anal.*, 1912, **17**, 47-50.)—In the following method the errors caused by the solubility of glycyrrhizic acid in water (about 0·5 per cent.) are eliminated: Two grms. of the sample are treated with 50 c.c. of hot water, the solution filtered through a tared filter, and the insoluble organic matter washed with 50 c.c. of hot water, dried, and weighed. The filtrate is treated with 5 c.c. of N-sulphuric acid and allowed to stand for about twelve hours, after which the supernatant liquid is decanted, and the precipitated glycyrrhizic acid washed with as little water as possible until the washings are no longer acid. The precipitate is now dissolved in ammonia solution, and the resulting solution will contain the bulk of the glycyrrhizic acid. The filtrate and the washings are then evaporated nearly to dryness, the black viscous residue is mixed with successive portions of 10, 10, and 5 c.c. of water, and the liquids filtered after each treatment. The fresh quantity of glycyrrhizic acid thus separated is dissolved in ammonia solution and added to the main solution, the whole being then evaporated to dryness, and the residue of ammonium glycyrrhizate dried at 100° C. until constant in weight. The gummy substances may be estimated by evaporating the last filtrate to dryness, drying the residue at 100° C. until constant in weight, and deducting 0·330 grm. for the ammonium sulphate corresponding to the 5 c.c. of N-sulphuric acid originally added. As thus separated the gummy substances should be a transparent yellow mass, which, when dissolved and treated with an acid, should not yield more than a trace of glycyrrhizic acid. In various estimations the amount of glycyrrhizic acid retained by the gums did not exceed 0·010 grm., corresponding to a negligible error of not more than 0·5 per cent. in the amount of ammonium glycyrrhizate in the sample. (*Cf.* ANALYST, 1911, **36**, 409, 410, 497.) C. A. M.

Bitter Resins of Hops in Worts and Beers and their Volumetric Estimation. D. Neumann. (*Wochensch. Brau.*, 1912, **29**, 97-99.)—In a previous note (ANALYST, 1912, 92) the author described how the resins could be extracted from wort or beer by means of ether without the production of troublesome emulsions. The beer is shaken, in small portions at a time, with five times its volume of ether, the same ether being used for each successive portion of wort or beer until the entire sample has been treated. Each extraction should be performed in a shaking machine for at least fifteen minutes. The ethereal extract is concentrated to a volume of 15 to 20 c.c.; it is treated with 40 to 50 c.c. of benzene, and transferred to a separating funnel, in which it is washed with water until the washings are neutral. Too vigorous shaking at this stage is to be avoided. Finally, the ethereal benzene solution of the resins is titrated with $\frac{N}{10}$ alcoholic potassium hydroxide according to the usual method of hop analysis. The error, due to the carbon dioxide of the air, using phenolphthaleïn, amounts to about 0·1 c.c. of $\frac{N}{10}$ potash, or 0·004 grm. of bitter resin. Performed in conjunction with a blank test, this method appears to give very satisfactory results when applied to unhopped worts, to which known quantities of standardised hop-extract were added. But when the worts were boiled for two hours under a reflux condenser with the hop resins, a considerable deficiency, corresponding to about 35 per cent. of the original resin, was found on analysis. This deficiency is not due to loss of resin by volatilisation or precipitation, but evidently corresponds to a constitutional change in the resin itself. Experiments with the purified crystalline α-resin of hops showed that this substance, after boiling with wort, undergoes a change, whereby a portion of it becomes soluble in water without losing its bitter properties, and is removed in the washing process. The author has applied his method to the analysis of ordinary beers (1 litre taken for extraction), and found that it gave concordant results with beers of the same manufacture, and that it showed clearly the effect of variations in the proportions of hops used in brewing. J. F. B.

Examination of Meal. E. Kohn. (*Chem. Zeit.*, 1912, **36**, 121-123.)—In the microscopical examination of meals for the detection of foreign seeds, the author prepares the sample in the following manner: 0·5 grm. of the meal is shaken in a test-tube with 10 c.c. of ether, and the mixture is poured out into a wide, shallow porcelain dish. The characteristic seed-hairs for the identification of the meal constituents can easily be floated off from the surface of the ether in the test-tube, whilst yellow-coloured bran particles and foreign impurities can be separated by tilting the dish so as to spread the liquid in a thin layer as the ether evaporates. Alternatively, the ethereal-meal suspension may be poured on to filter-paper and allowed to spread out.

A method for the differentiation of various meals may be based on the variable rates of the attack on the starch granules by diastase. Two grms. of starch or meal are weighed out into a 300 c.c. Erlenmeyer flask, mixed with 100 c.c. of water, 0·4 grm. of Merck's diastase, and 1 c.c. of $\frac{N}{10}$ hydrochloric acid. The mixture is digested at the optimum temperature (50° to 52° C.) for a given time—*e.g.*, four hours. The liquid is filtered, and the amount of dissolved solids or sugar is determined with the pyknometer, or preferably by Fehling's solution. The figures so obtained are compared with those established with standard meals. Rye starch or meal gives the highest values, bean flour the lowest; wheat flour shows higher values than barley. The values vary slightly for different grades of the same flour; mixtures show intermediate values.

J. F. B.

Estimation of Potassium Nitrate in Meats. J. Tillmans and A. Splittgerber. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 49-56.)—When the quantity of potassium nitrate in a sample of meat does not exceed 1·5 per cent., it may be accurately estimated by Noll's brucine-sulphuric acid method, or by the diphenylamine-sulphuric acid method. In order to obtain a solution to which the reagents may be applied, the following procedure is recommended: Fifty grms. of the finely-divided meat are immersed in about 500 c.c. of water for three hours, with occasional shaking; the aqueous portion is then decanted through a filter and diluted to a volume of 1 litre. Fifty c.c. of this solution are now treated with 50 c.c. of a mixture consisting of equal volumes of 5 per cent. mercuric chloride solution and 2 per cent. hydrochloric acid, and, after filtration, the nitrate is estimated in a definite volume of the filtrate by means of the methods mentioned; the comparison solutions employed should contain definite quantities of potassium nitrate, and, in the case of the diphenylamine method, sodium chloride must be added to these solutions as well as to the meat solution (*cf.* ANALYST, 1911, **36**, 67). The brucine-sulphuric acid method will be found to be the more convenient for single estimations, and the diphenylamine method preferable when a series have to be made. In cases where the amount of potassium nitrate present in the meat exceeds 1·5 per cent., Schloesing's method should be employed. The results of a considerable number of analyses are given, which show that the methods are trustworthy. The preparation of the diphenylamine reagent has been described previously (*loc. cit.*), and the brucine reagent is made by moistening 0·25 grm. of brucine with water, and then dissolving it in 100 c.c. of concentrated sulphuric acid.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Dextrose in Urine and Blood. B. Oppler. (*Zeitsch. physiol. Chem.*, 1911, **75**, 71-134; through *Chem. Zentralbl.*, 1912, I., 380-381.)—For the estimation of dextrose at concentrations below the limits at which the qualitative tests are sensitive, the urine is decolorised by the addition of crystallised phosphotungstic acid, protected from the light. After standing for twelve hours, the liquid is filtered, and the filtrate treated with neutral lead acetate; after a further twelve hours in the dark it is again filtered, and the lead is removed by hydrogen sulphide. Urines thus treated in most cases no longer contain any perceptible quantity of lævo-rotatory substances; at the same time, substances which interfere with the cupric reducing reaction are removed. The dextrose can then be estimated by polarisation, or cupric reduction, before and after fermentation by yeast. Healthy urine contains, however, cupric reducing, optically inactive substances, which are removable by yeast, to an amount equivalent to 0·04 per cent. of dextrose. In the urine of mental patients dextrose was found in the majority of cases, but mostly in small amounts. Such urine frequently shows an abnormally high lævo-rotation, equivalent to as much as 0·97 per cent. of glucose. The application of the above method of purification to blood showed that the colouring, lævo-rotatory, and interfering matters of the serum behaved very much in the same way as those present in urine, and the methods applicable for the estimation of sugar in urine are likewise suitable in the case of blood. J. F. B.

Estimation of Sugar in Blood. D. Takahashi. (*Biochem. Zeitsch.*, 1911, **37**, 30-33.)—Results of estimations of sugar in dogs' blood by various methods are recorded; the methods described by Bertrand and by Kumagawa and Suto yielded results which agreed with one another and with those obtained by the polarimetric method. The total amount of sugar found to be present varied from 0·12 to 0·18 per cent. when these methods were employed, whilst Bang's method gave results which were about 0·03 per cent. higher. No sugar was found after fermentation.

W. P. S.

Are the Alkalinity and Peroxydase of Milk Identical? W. D. Kooper. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 1-13.)—The alkalinity value of milk (as measured by the quantity of acid required to change the violet coloration to green when 100 c.c. of milk are treated with a mixture containing *p*-phenylenediamine hydrochloride, guaiacum, and hydrogen peroxide, the alkalinity value being expressed in c.c. of $\frac{N}{10}$ acid) is not a constant quantity, varying from 106 to 112, with an average of 110. The value is higher for colostrum, but decreases as the lactation period proceeds; for fresh goats' milk it generally lies above 120. The addition of water to milk raises the value, whilst the acidity of the milk is diminished, and the decrease in the acidity is greater in the case of old milk than with fresh milk; on the contrary, the increase in the alkalinity is smaller. These and other results of an investigation carried out by the author led him to the opinion that the property possessed by milk of being capable of producing a coloration with the above-mentioned reagent is due to the action of inorganic compounds contained in the milk. These compounds appear to be various phosphates of the alkalis and alkali-earths. W. P. S.

Separation of Rennin and Pepsin by Means of the Electric Current. W. E. Burge. (*Amer. J. Physiol.*, 1912, **29**, 330-334; through *Chem. Zentralbl.*, 1912, I., 687.)—When an electric current of 10 milliampères was passed through a solution containing rennin and pepsin for twenty-four hours, the liquid completely lost its property of digesting fibrin, whereas the activity of the rennin, as displayed by its power of coagulating fresh milk, remained unchanged. J. F. B.

Estimation of the Acidity of Urine. L. Grimbert and J. Morel. (*Comptes rend.*, 1912, **154**, 378-380.)—In titrating the acidity of urine, with phenolphthaleïn as indicator, apparent neutrality is obtained when the monosodium phosphate has been converted into disodium phosphate, but the *absolute* acidity would correspond to the formation of trisodium phosphate. If the conversion of the monosodium salt into the disodium salt be regarded as indicating the *real* acidity, account must still be taken of the influence of ammonium and calcium salts upon the results of the titration. The first source of error may be obviated by titrating the ammonia by Ronchèse's formaldehyde method, and making a correction from the result obtained; and the second by adding to the urine a small quantity of powdered potassium oxalate to remove the calcium salts. If, then, the proportion of phosphoric acid in the urine $\left(=\frac{P_2O_5}{2}\right)$ be calculated into that of the corresponding monosodium salt, the acidity of the latter towards phenolphthaleïn will represent the phosphatic acidity, p, of the urine. On the other hand, if R represent the real acidity as estimated by direct titration, it is possible to calculate: (1) the organic acidity, a; (2) the absolute acidity, A, and the amounts of monosodium and disodium phosphate in the urine. If the phosphatic acidity is inferior to the real acidity, disodium phosphate cannot be present, but there will be organic acidity. Since $a = R - p$, and $A = 2p + a$, the proportion of monosodium phosphate will be equivalent to $p \times 3{\cdot}680$. Again, if the phosphatic acidity is superior to the real acidity, there can be no organic acidity, but the total phosphatic acidity must be distributed between the acidity due to the monosodium phosphate ($= m$), and that due to the disodium phosphate, d. The calculations would then be as follows: $m = R$, $d = p - R$, $A = 2R + d$, whence—

$$NaH_2PO_4 = R \times 3{\cdot}680, \text{ and}$$
$$Na_2HPO_4 = d \times 4{\cdot}356.$$

C. A. M.

ORGANIC ANALYSIS.

Alkaloidal Assay of Calabar Beans. A. H. Salway. (*Amer. J. Pharm.*, 1912, **84**, 49-51.)—The United States Pharmacopœia method for this assay was found by the author to give very low results, and the following modification is suggested: Twenty grms. of powdered beans (No. 60 powder) are well shaken with 200 c.c. of ether, 10 c.c. of an aqueous solution of sodium carbonate (10 per cent.) are added, and the mixture shaken vigorously at intervals for four hours. The powder is allowed to settle, after which 100 c.c. of the ethereal liquid are transferred to a separator and sufficient $\frac{N}{10}$ sulphuric acid added to make it distinctly acid. After thorough shaking, the acid layer is drawn off, and the operation repeated twice, using 10 c.c. of $\frac{N}{10}$ sulphuric acid each time. The combined acid extracts are made alkaline with sodium carbonate solution (10 per cent.), and the liquid extracted successively with 10 quantities of 20 c.c. each of ether. After washing the whole ethereal extract with 5 c.c. of water, the ether is distilled, the alkaloidal residue dissolved in 5 c.c. of $\frac{N}{10}$ sulphuric acid, and the excess of the latter titrated with $\frac{N}{50}$ alkali, using iodeosin as indicator.

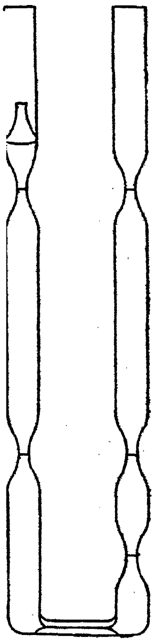
A. R. T.

Application of Methyl-Red to the Colorimetric Estimation of Hydrogen Ion Concentrations. S. Palitzsch. (*Biochem. Zeitsch.*, 1911, **37**, 131-138.)—Methyl-red (*p*-dimethylaminoazobenzene-*o*-carboxylic acid) is particularly suitable for use as an indicator in the measurement of hydrogen ion concentrations between the zones $p_H = 4{\cdot}2$ and $p_H = 6{\cdot}3$, the change of colour being from violet-red to yellow.

A table of corrections is given, these corrections (which are only small) being applied in case proteins and salts are present. W. P. S.

Detection of Pine Oil in Turpentine Oil. C. Piest. (*Chem. Zeit.*, 1912, **36**, 198.)—A mixture of 5 c.c. of the turpentine oil and 5 c.c. of acetic anhydride is shaken with 10 drops of concentrated hydrochloric acid, and allowed to cool, after which an additional 10 drops of hydrochloric acid are added, and the mixture again shaken. Under these conditions pure turpentine oil remains transparent and light yellow, whilst pine oil becomes black. This test will give a distinct coloration in the presence of 10 per cent. of pine oil, whilst even 5 per cent. causes a slight darkening of the liquid. In the case of old turpentine oil the sample should be distilled, and the test applied to the distillate. Purified pine oil also gives a dark coloration, though not to so marked an extent as ordinary pine oil. C. A. M.

Viscosity of Fish Oils. G. F. White. (*J. Ind. Eng. Chem.*, 1912, **4**, 106-110.)—For determining the viscosity of moderately viscous liquids such as fish oils, the author has devised a viscosimeter very similar to that previously described by him for less viscous liquids (*Biochem. Zeitsch.*, 1911, **37**, 482), but with a shorter capillary and with the volumes of the limbs considerably less. The method of standardising the instrument was similar to that described in detail in the earlier paper, but water could not be used as the time of flow would have been too short. Therefore, the viscosity of a sample of Menhaden oil was determined in the earlier form of apparatus which had been standardised with water, the temperature being 50° C. to reduce the time necessary for measurement. The viscosity was found to be 0·1518 in the left limb and 0·1514 in the right limb. The new viscosimeter was next calibrated with this oil and the following constants obtained for the instrument:



Length of capillary (approximate) ...	1·7 cm.
Volume of left limb	2·1310 c.c.
Volume of right limb	2·0748 c.c.
Pressure correction, left limb	1·23 cm.
Pressure correction, right limb	1·20 cm.
Ratio of r^4 to l	0·00004577. 0·00004582.

In use the liquid is forced through the capillary under a pressure of 100 to 180 cm. The kinetic energy correction $\frac{vd}{8\pi tl}$ in the formula $\eta = \frac{\pi r^4 pt}{8vl} - \frac{vd}{8\pi tl}$ is low, this being necessary if the work is to be of a high order of accuracy. This correction was always less than 0·5 per cent. of the total viscosity. In the above equation η is the viscosity, r and l are the radius and length of the capillary, v is the volume of the liquid of density d, p is the pressure, and t is the time of flow.

With this instrument the viscosity of the following oils in absolute measure (c.g.s. units) was determined at 30°, 50°, 70°, and 90° C.:

	30°.	50°.	70°.	90°.
Menhaden I. (*Brevoortia tyrannus*) ...	0·2961	0·1516	0·09555	0·06350
,, II., dark	0·6019	0·2935	0·1683	0·10970
,, III., light	0·3238	0·1727	0·1049	0·07097
Whale (*Balæna mysticetus*)	0·3485	0·1822	0·1101	0·07241
Cod liver (*Gadus callarias*), white ...	0·3884	0·2003	0·1188	0·07872
,, ,, brown ...	0·3924	0·2079	0·1195	0·07855
Sperm (*Physeter macrocephalus*) ...	0·3333	0·1720	0·1038	0·06655
Dogfish (*Mustelus canis*)	0·4319	0·2135	0·1261	0·08283
,, liver, I.	0·4096	0·2072	0·1212	0·07972
,, ,, II.	0·3762	0·1977	0·1161	0·07740
,, ,, III.	0·3776	0·1943	0·1154	0·07638
Spiny dogfish liver (*Squalus acanthias*)	0·3993	0·2049	0·1214	0·07957
Sand shark liver (*Cacharias littoralis*)	0·3678	0·1943	0·1177	0·07916
Hammer-head shark liver (*Sphyrna zygæna*	0·5470	0·2681	0·1537	0·09794
Torpedo liver I. (*Tetranarce occidentalis*)	0·4263	0·2133	0·1257	0·08073
,, II.	0·4774	0·2356	0·1379	0·08751
Squeteague (*Cynoscion regalis*) ...	0·4980	0·2402	0·1384	0·08099
Sarp (*Stenotomus chrysops*)	0·4195	0·2141	0·1239	0·08124
Butter-fish (*Poronotus tricanthus*) ...	0·4266	0·2092	0·1194	0·07613
Eel (*Anguilla rostrata*)	0·4015	0·2026	0·1187	0·07803

When, after heating to 90° C., these oils were cooled to 30° C. and their viscosity redetermined at that temperature, this was found to have been unaffected by the heating. The fluidities of the oils are not linear functions of the temperature, indicating that there is some association at the lower temperature (*cf.* Bingham, *Zeitsch. Phys. Chem.*, 1909, **66**, 1). G. C. J.

"Pergamyn" and Imitation Parchment Papers. W. Herzberg. (*Mitt. K. Materialprufungsamt*, 1911, **29**, 464-467; through *Chem. Zentralbl.*, 1912, I. 688.) —"Pergamyn," "greaseproof," or imitation parchment papers owe their special qualities to the mechanical treatment followed in their manufacture and not to any addition of an external substance, such as paraffin wax. Genuine parchment papers possess somewhat similar characters owing to a chemical treatment, a modification of the fibres by the action of sulphuric acid. Customs officials are not always in a position to recognise these distinctions, and pergamyn papers and the like are sometimes charged as impregnated or waxed papers, sometimes as genuine parchment papers.

Test for Paraffin Wax.—The paper is extracted with ether, the ethereal extract is saponified with alcoholic potash, the dried residue after saponification is extracted with ether, and the ethereal extract is dissolved in hot acetic anhydride. If paraffin wax is present, it separates out on cooling. It is generally sufficient to observe whether the paper loses its lustre and transparency after extraction with ether; pergamyn papers remain unaltered.

Test for Genuine Parchment Papers.—A portion of the paper is boiled with a 2 or 3 per cent. solution of sodium hydroxide whilst stirring. Imitation parchment papers fall to pieces under this treatment, whilst genuine, chemically modified parchment papers retain their form. J. F. B.

Unification of Reducing Sugar Methods (A Correction for Lactose). P. H. Walker.

(*J. Amer. Chem. Soc.*, 1912, **34**, 202-209.)—Attention has been called to arithmetical errors in the tables originally published for lactose (*J. Amer. Chem. Soc.*, 1907, **29**, 541). Table IV. giving the amount of sugar for each mgrm. of cuprous oxide has been wrongly calculated and does not agree with the experimental results of Table III., which is correct. The same error has been reproduced in the U. S. Bulletin, No. 107, Bureau of Chemistry, Official and Provisional Methods of Analysis, pp. 243-251, which has been in use for several years. In the formula—

$$y = a + bx + cx^2,$$

where y = cuprous oxide and x = reducing sugar, the true values in the case of lactose are $a = 4{\cdot}1759$, $b = 1{\cdot}48697$, $c = -0{,}00009$. The lactose used was the crystallised hydrate $C_{12}H_{22}O_{11} + \frac{1}{2}H_2O$. J. F. B.

Estimation of Sugars in Natural Products. C. Neuberg and M. Ishida.

(*Biochem. Zeitsch.*, 1912, **37**, 141-169.)—It is shown that by treating sugar solutions with 50 per cent. mercuric acetate solution followed by precipitation with 25 per cent. phosphotungstic acid solution, all optically active proteins, their derivatives (amino-acids, etc.), and other substances are removed, whilst the sugars remain in solution and can be estimated polarimetrically. The process is particularly applicable to the analysis of mixtures of sugar with amino-acids, molasses, etc., and results of experiments are recorded showing that the method is trustworthy. The amino-sugar, *d*-glycosamine, may be estimated in the presence of amino-acids by means of this method, as it is not precipitated by the reagents mentioned. A method is described for preparing the phosphotungstic acid reagent; 200 grms. of crystallised sodium tungstate are dissolved in hot water, 5 grms. of solid phosphoric acid are added, and the clear solution is mixed first with 60 c.c. of dilute sulphuric acid, and then with 58 grms. of concentrated sulphuric acid, the solution being cooled during the addition of the acid. The solution is next evaporated on a water-bath to a volume of 290 c.c., cooled in an ice-chest, the crystals of sodium sulphate are separated, and the residual solution is diluted with an equal volume of water. W. P. S.

INORGANIC ANALYSIS.

Detection of Bromine by Fluoresceïn. H. Baubigny.

(*Bull. Soc. Chim.*, 1912, **11**, 13.)—The author's test (*Comptes rend.*, 1897, **125**, 654) has been stated by Pribram, and later by Labat, to be inconclusive as applied to the detection of bromine in animal organs, on the ground that chlorine or iodine would give the same reaction. This is denied: the chlorine compound of fluoresceïn is almost colourless, and iodine should be converted into iodate by oxidation with permanganate, as indicated in the original paper. C. E. M.

Estimation of Alkalis in Silicates by Fusion with Calcium Chloride. E. Mäkinen. (*Zeitsch. anorg. Chem*, 1912, **74**, 74-78.)—Instead of the calcium carbonate and ammonium chloride of the Lawrence Smith method (*Ann. Chem. u. Pharm.*, 1871, **159**, 82), anhydrous calcium chloride is recommended. The calcium chloride should be prepared in the laboratory, as the purest commercial salt may contain as much as 0·02 grm. of alkali chloride in 7 grms., the amount of flux recommended for use with 0·5 grm. of the silicate. Intimate admixture of the silicate with the flux is unnecessary, as the latter, unlike the mixture of Lawrence Smith, really melts. The crucible is supported on asbestos millboard, so that its upper portion and the cover may remain relatively cool and so serve to prevent volatilisation of alkali chloride. The crucible is heated gently at first until the calcium chloride is fused, but is then raised to a full red heat by means of a Teclu burner. The use of a blowpipe-flame leads to volatilisation of alkali. After twenty-five to thirty minutes fusion the flame is withdrawn, the crucible cooled suddenly, and the analysis completed in the usual manner, except that the use of ammonium carbonate in place of ammonium oxalate is recommended for removing the last traces of calcium. Ammonium carbonate is said to separate the last traces of calcium as well as oxalate, and is preferred because of the high temperature which is necessary to expel oxalic acid from the residue of alkali chlorides. When the results obtained differ from those yielded by the Lawrence Smith method, they are higher, and the author thinks they should be preferred. G. C. J.

Estimation of Ferrous Iron in Silicate Rocks. M. Dittrich and A. Leonhard. (*Zeitsch. anorg. Chem.*, 1912, **74**, 21-32.)—The paper contains a detailed description of the experiments which led Dittrich to publish the statement (Analyst, 1911, **36**, 602) that the uncertain end-point in the titration of ferrous iron in certain rocks, after decomposition by means of hydrofluoric acid, was due to the presence in those rocks of Ti_2O_3, and not to the presence of manganese. The following method of conducting the estimation of ferrous iron avoids any difficulty due to the presence of Ti_2O_3. From 0·8 to 1 grm. of the not too finely powdered sample is mixed with 0·5 grm. quartz sand (Analyst, 1911, **36**, 372) and 2 c.c. sulphuric acid, and decomposed by means of 8 c.c. of hydrofluoric acid. The crucible is placed on a boiling water-bath and covered with an inverted funnel, which is sufficient to prevent any appreciable oxidation during the few minutes required for decomposition of the rock. The contents of the crucible are poured, without previous cooling, into a mixture of 100 c.c. water, 10 grms. precipitated silica, 20 grms. potassium sulphate, and 2 c.c. sulphuric acid, and the mixture is titrated at once with permanganate. G. C. J.

Estimation of Chromium and its Separation from Vanadium Steels. J. R. Cain. (*J. Ind. Eng. Chem.*, 1912, **4**, 17-19.)—After drawing attention to the difficulties which attend the estimation of chromium in chrome-vanadium steels, the author recommends the following method, which depends on the fact established by him (Analyst, 1911, **36**, 475) that chromium can be precipitated completely by boiling the nearly neutralised (ferrous) solution with barium carbonate, and on the observation of Noyes and Bray (*Technology Quarterly*, 1908, **21**, 14), that chromate may be separated from vanadate by precipitation as lead chromate if certain precautions are observed.

The drillings, which should preferably contain not more than 0·06 or 0·07 grm. of chromium, are placed in a 300 c.c. Erlenmeyer flask and dissolved in fuming hydrochloric acid of which about 10 c.c. is taken for each grm. of steel. The solution is diluted to 100 or 150 c.c. with hot water and nearly neutralised with saturated sodium carbonate solution. A slight excess of an emulsion of barium carbonate is added and the mixture boiled vigorously for ten or fifteen minutes with small additions of barium carbonate every two or three minutes, the flask being kept covered to exclude air as completely as possible. The excess of barium carbonate used should not be more than 2 grms. or there may be difficulty in extracting the chromium by a single fusion. The precipitate is allowed to settle and is then filtered off and washed twice with hot water, these operations being conducted as quickly as possible. The filter and precipitate are transferred to a platinum crucible, the paper is burned away, 2 grms. sodium carbonate and about 0·25 grm. potassium nitrate are mixed in, and the mixture is maintained in a state of fusion for twenty minutes. The product of the fusion is digested with boiling water and the solution filtered. Hydrogen peroxide (1 or 2 c.c. of a 3 per cent. solution) is added to destroy any nitrite, and the solution is boiled for five minutes to decompose the excess of peroxide. The solution is cooled and transferred to a separator, rendered slightly acid with nitric acid and vigorously shaken to expel carbon dioxide. It is then transferred to a beaker, just neutralised with sodium hydroxide, and then made acid by addition of 2 c.c. nitric acid (1 : 1) for each 100 c.c. of solution. Lead nitrate (20 c.c. of a 20 per cent. solution) is added to the cold solution with vigorous stirring, and the lead chromate is filtered off on asbestos and washed three or four times with cold water. The asbestos mat is transferred to a beaker or flask and the lead chromate decomposed with hot hydrochloric acid (1 : 4). The solution is cooled, made up to 150 or 200 c.c., and titrated against $\frac{N}{10}$ ferrous sulphate with ferricyanide as an outside indicator. The amount of sodium carbonate permissible in the fusion is limited by the fact that for a successful separation of chromium from vanadium not too much sodium nitrate should be present (Noyes and Bray, *loc. cit.*), and it is this necessity for restricting the amount of sodium carbonate which makes it advisable to take for analysis not more of the sample than corresponds to 6 or 7 cgrms. of chromium. Larger amounts are not readily extracted by 2 grms. of sodium carbonate, especially if too great an excess of barium is present. The shaking of the solution to eliminate carbon dioxide determines the subsequent formation of a precipitate which settles and filters well, and equally cogent grounds underlie other directions as to manipulative detail. Test numbers, obtained with solutions of a vanadium and chromium free steel to which known amounts of bichromate and vanadate were added, show maximum errors of ± 0·0004 grm. with from 0·02 to 0·3 grm. chromium present. The filtrates from the barium carbonate precipitation were tested for chromium with negative results in every case, and in only a few cases was any trace of vanadium found in the chromium solution after titration, and in those cases the amount was negligibly small. On the other hand, it is well to examine the insoluble matter from the fusion by fusing it again with sodium carbonate and potassium nitrate. With much chromium in the original weight of drillings, a yellow-coloured solution may result. If so, the chromium in it may be estimated with sufficient exactness colorimetrically. G. C. J.

Analysis of Higher Oxides of Lead. A. Chwala and E. Colle. (*Gazz. Chim. Ital.*, 1911, **41**, 551-579.)—From the results of an experimental study of the different methods proposed for the analysis of lead peroxide the author recommends the following modification of Lux's method (*Zeit. anal. Chem.*, **19**, 153) as the most convenient and reliable: From 1 to 2 grms. of the sample are treated with 100 c.c. of $\frac{N}{1}$ nitric acid and 25 c.c. of $\frac{N}{2}$ oxalic acid solution, and the liquid boiled for ten to fifteen minutes, until solution is complete. The excess of oxalic acid is then titrated with standard permanganate solution, and the amount of lead peroxide calculated from the result—

$$PbO_2 + C_2H_2O_4 = PbO + H_2O + 2CO_2$$

The duration of the boiling with nitric acid and the concentration of that acid have considerable influence upon the results. The results of experiments have shown that the differences between the results given by the oxalic acid method and the iodometric methods are entirely due to the occurrence of secondary reactions in the latter, leading to the formation of lead iodide and methyl acetate, so that part of the iodine escapes titration—

$$Pb(CH_3COO)_2 + 2I = CH_3COOCH_3 + PbI_2 + CO_2$$

In some instances the error thus produced may be considerable, and, in any case, should always be taken into account in iodometric estimations of oxidising substances in the presence of weak acids and heavy metals. Various samples of commercial lead peroxide analysed by the foregoing method contained from 43·5 to 95·56 per cent. of lead peroxide. C. A. M.

Bismuthate Method for Manganese. D. J. Demorest. (*J. Ind. Eng. Chem.*, 1912, **4**, 19.)—The following modification of the bismuthate method is recommended for use with chrome and chrome-vanadium steels. The drillings are dissolved in nitric acid (sp. gr. 1·1), boiled to expel nitrous fumes, and potassium bismuthate is added until permanganic acid or manganese dioxide persists on boiling. Potassium nitrite is added to dissolve the manganese dioxide, and the solution is boiled to expel nitrous fumes, after which it is cooled and the manganese oxidised by bismuthate in the cold as usual. The solution is then filtered rapidly through asbestos on glass wool and titrated with a standard solution of sodium arsenite, made by dissolving 2·25 grms. of arsenious acid in a hot solution of sodium carbonate and diluting to 2·5 litres. The test numbers, obtained in presence of three or four times as much chromium or vanadium as manganese, are good, the worst being 0·671 per cent. found with 0·666 present. G. C. J.

Determination of the Density of Minerals by Means of Rohrbach Solution of Standard Refractive Index. H. E. Merwin. (*Amer. J. Science, Silliman*, 1911 [4], **32**, 425; through *Chem. Zentralbl.*, 1912, I., 524.)—The density of a solution contained in the cell of a refractometer is adjusted until a fragment of the mineral under examination is just suspended in it; its refractive index is determined, and from this its density, and hence the density of the sample, is found. For Rohrbach (barium mercury iodide) solution, the relation between D (density) and n (refractive index), at 20° C., is as follows:

D =	3·449	3·396	3·246	3·180	3·046	2·980
n =	1·7686	1·7950	1·7312	1·7195	1·6944	1·6823
D =	2·748	2·649	2·648	2·367	2·163	2·067
n =	1·6391	1·6207	1·6205	1·5685	1·5320	1·5148

For densities between 2·25 and 3·40 it may be expressed, with a maximum error of ± 0·002, by the empirical formula:

$$D^{20} = 5{\cdot}39\,(n - 1{\cdot}5467) + 2{\cdot}25 = 5{\cdot}39n - 6{\cdot}0865.$$

For variations from 20° exceeding 3° a correction of ± 0·001 per ± 2° is necessary. For densities between 2·0 and 2·25, $D = 5{\cdot}7n - 6{\cdot}567$; and between 3·4 and 3·5, $D = 5{\cdot}52n - 6{\cdot}313$. For approximations, the mineral is brought to suspension, and n then determined on a drop of the liquid. O. E. M.

Estimation of Nitrates in Sewage Effluents. H. Silvester.

(*J. Soc. Chem. Ind.*, 1912, **31**, 95-96.)—Effluents derived from sewage containing waste gas liquor may contain thiocyanates, and in presence of thiocyanates the phenol-sulphonic acid method for the estimation of nitrates is not trustworthy. The zinc-copper couple method is recommended as the best for sewage effluents. In using this method it is convenient to have an approximate idea of the amount of nitrate present, and for this purpose the diphenylamine test serves well. To 2 c.c. of the effluent a few drops of the diphenylamine reagent and 10 c.c. of sulphuric acid (free from nitrate) are added. The intensity of the blue coloration which results enables the analyst to form a rough estimate of the amount of nitrate present.

G. C. J.

Detection of Nitrates in Presence of Nitrites. H. K. Sen and B. B. Dey.

(*Zeitsch. anorg. Chem.*, 1912, **74**, 52-54.)—When nitrites are destroyed by means of urea, as in Piccini's method for the detection of nitrate in presence of nitrite, small quantities of nitrate are invariably formed by the decomposition of nitrous acid into water, nitric oxide, and nitric acid. It is shown that the reaction of hydrazine sulphate with nitrites (ANALYST, 1911, **36**, 468) is unattended by the formation of even such traces of nitrate as may be detected by the diphenylamine reaction, and hydrazine sulphate is therefore recommended for the preliminary destruction of nitrite when it is wished to test for nitrate in a liquid containing nitrites.

G. C. J.

Note by Abstractor.—Stutzer and Goy (ANALYST, 1911, **36**, 524) have stated that the decomposition of nitrite by hydrazine sulphate is never complete.

Estimation of Phosphoric Acid by von Lorenz's Method. H. Neubauer and F. Lücker.

(*Zeitsch. anal. Chem.*, 1912, **51**, 161-175.)—The following procedure is recommended in estimating phosphoric acid by this method, the principal modification introduced being that acetone is employed instead of alcohol and ether for washing the phosphomolybdate precipitate. The reagents required are: (1) *Molybdic acid reagent.* One hundred and fifty grms. of ammonium molybdate are dissolved in 400 c.c. of boiling water, and the solution, when cold, is added gradually to a solution of 50 grms. of ammonium sulphate in 450 c.c. of nitric acid of sp. gr. 1·4; the whole is then diluted to a volume of 1 litre, filtered, and stored in a dark place. (2) *Nitric acid containing sulphuric acid.* Thirty c.c. of sulphuric acid of sp. gr. 1·84 are added to 1 litre of nitric acid of sp. gr. 1·20. (3) Nitric acid of sp. gr. 1·20. (4) A 2 per cent ammonium nitrate solution. (5) Neutral acetone. A quantity of from 10 to 15 c.c. of the phosphoric acid solution under examination (for instance, the solutions obtained in the analysis of fertilisers for total and citric acid soluble phosphoric acid) is diluted to a volume of 50 c.c.; should sulphates not be present, the dilution is made with the nitric acid containing sulphuric acid, otherwise with the dilute nitric acid. The mixture is then heated nearly to boiling, and 50 c.c. of the molybdate reagent are added, the whole being stirred for five minutes. After the lapse of from two to eighteen hours (the longer period is necessary if less than 3 mgrms. of phosphoric be present), the precipitate is collected in a Gooch crucible, washed with the 2 per cent. ammonium nitrate solution about four times, and then twice with acetone. The crucible is now placed for at least thirty minutes in a vessel in which the pressure has been reduced below 150 mm., and then weighed. The precipitate thus obtained contains 3·295 per cent. of phosphoric anhydride (P_2O_5). It is shown that the acetone removes the ammonium nitrate completely from the precipitate and filter, and results of estimations are recorded which show that the method is accurate. W. P. S.

Determination of the Penetration of Salts in Treated Wood. (*Forest Service Circular*, 190, *U.S. Dept. of Agriculture;* through *Chem. Trade Journal*, 1912, **50**, 12.)—If zinc chloride is the preservative, a representative disk of the wood is dipped first, for not more than ten seconds, into 1 per cent. potassium ferrocyanide, and then, after removal of the excess with filter paper, into 10 per cent. uranium acetate; it is then allowed to dry. The untreated portion will now be dark red, the treated portion somewhat lighter than the natural wood. The method allows 0·2 pound per cubic foot to be detected. Copper and iron salts are determined by the dark-red and deep blue coloration, respectively, of the treated zone on immersion in potassium ferrocyanide solution; mercuric chloride by the blackening in hydrogen sulphide solution. O. E. M.

Rapid Method for Determination of Sulphur in Roasted Blende. C. C. Nitchie. (*J. Ind. Eng. Chem.* 1912, 4, 30-32.)—For determining whether a particular car-load of roasted blende is sufficiently roasted to be suitable for use in the spelter furnaces, or whether it should be returned to the kiln for additional roasting, a rapid method is essential. The following method is recommended as rapid, and sufficiently exact for the purpose. The sample is ground no more finely than is necessary to get a 1-grm. sample which shall be fairly representative; finer grinding prolongs the time necessary for the subsequent roasting of the ore. One grm. of the sample is weighed out into a boat, which is placed in a silica combustion-tube previously heated to 1000° C., and a current of air, freed from carbon dioxide, is led through the tube and through an absorption apparatus containing standard caustic alkali. A fume of zinc oxide, formed by reduction of zinc oxide by zinc sulphide, volatilisation of the zinc, and its subsequent oxidation in the air-current, passes through the absorption apparatus, only negligible traces being dissolved by the alkali solution. Experiment has shown that the roasting may be discontinued when this fume is no longer seen in the absorption apparatus. The only sulphur then remaining with the roasted ore is that combined as calcium sulphate. The whole of the "false" sulphur—sulphur which should have been eliminated in the kiln, but which remained as sulphide or sulphate of zinc or lead in the sample—will have passed into the absorption apparatus, mostly as sulphur dioxide with some trioxide. The excess of alkali hydroxide is titrated with standard acid, using phenolphthaleïn as indicator, and the sulphur in 1 grm. of the sample is calculated from the alkali neutralised by the gases resulting from roasting the ore. The roasting process usually occupies only about six minutes. The maximum error of the method is −0·1 per cent. on the ore, which is quite near enough for the purpose, the percentage of "false" sulphur in roasted blende being of the order of 1 per cent. Without modification the method is not sufficiently exact for application to unroasted blende.

G. C. J.

Rapid Volumetric Method for Estimation of Free Sulphur. C. Davis and J. L. Foucar. (*J. Soc. Chem. Ind.*, 1912, **31**, 100.)—The method depends on the quantitative conversion of the sulphur into thiocyanate by digestion with an alcoholic solution of sodium cyanide, and the subsequent titration of the thiocyanate. One grm. of the finely ground (60 mesh) spent oxide or other material is dried for an hour in the steam oven and then transferred, with 1·5 grm. sodium cyanide and 50 c.c. absolute alcohol, to a 250 c.c. flask. The contents of the flask are boiled under a reflux condenser for two hours, and the alcohol is then completely removed by distillation. The residue is dissolved in 100 c.c. of hot water, and the solution, when cool, made up to 250 c.c. To 25 c.c. of this solution, 75 c.c. of water and 5 c.c. of a saturated solution of iron alum are added and the mixture heated, with constant stirring, to 95° C., filtered, and the filter washed until free from thiocyanate. To the cool filtrate, 5 c.c. of nitric acid and enough water to bring the bulk up to about 500 c.c. are added, and the mixture is titrated with $\frac{N}{20}$ silver nitrate until the red colour of ferric thiocyanate disappears, or excess of silver nitrate may be added, and the solution titrated back with $\frac{N}{20}$ thiocyanate. Should the sample contain thiocyanate, as is most likely in the case of spent oxide, a blank test must be conducted, the thiocyanate being extracted with water. Test numbers obtained on applying the method to sulphur crystals agree well with those obtained by oxidising the sulphur and weighing as barium sulphate. The test numbers for spent oxides are from 0·3 to 0·4 per cent. lower than those obtained by oxidation of the carbon bisulphide extract, but the latter method includes as free sulphur the sulphur of any compound soluble in carbon bisulphide.

G. C. J.

Estimation of Sulphur in Illuminating Gas. J. Niermeyer. (*J. f. Gasbeleuchtung*, 1911, **54**, 1078-1079; through *Chem. Zentralbl.*, 1912, I., 375-376.)—According to Dennstedt and Ahrens, the ratio between sulphur dioxide and sulphur trioxide in the products of combustion of illuminating gas shows only small variations: 92 to 94 per cent. of the total sulphur exists as the dioxide and 6 to 8 per cent. as the trioxide. On this observation the author has based a simple method for the estimation of sulphur. A known volume of gas is burnt, the sulphur dioxide is estimated in the products, the quantity of sulphur found is increased by 6 to 8 per cent. and calculated per cub. m. of gas. The sulphur dioxide is absorbed in $\frac{N}{100}$ iodine solution, to which an excess of potassium iodide has been added to reduce the volatility of the iodine. The glass tube of a Drehschmidt sulphur-estimation apparatus is used, and the thin, downwardly inclined portion of the tube is utilised as a cooler for the products of combustion, which are then aspirated through the washing vessel containing the iodine. The gas-consumption is adjusted at 70 to 80 litres per hour; 10 c.c. of $\frac{N}{100}$ iodine solution, 10 c.c. of 10 per cent. potassium iodide solution, a few drops of starch solution, and 50 c.c. of water are placed in the washing vessel, and a current of air is aspirated. The burner is then placed under the glass tube, and, when the iodine solution is decolorised, the volume of gas burnt is read off on the meter. The quantity of iodine used represents 1·6 mgrm. of sulphur; this is divided by the number of litres of gas burnt, and the result is increased by 6 per cent., giving mgrms. of total sulphur per litre. J. F. B.

Estimation of Sulphur in Nitrocellulose. C. Kullgren. (*Zeitsch. ges. Schiess- und Sprengstoffwesen*, 1912, **7**, 89.)—About 0·75 grm. of the substance, denitrated by heating for thirty minutes on the water-bath with 10 c.c. hydrochloric acid, is dried in a large porcelain boat. It is then burnt in a stream of oxygen by the method of Barlow (*J. Amer. Chem. Soc.*, 1904, **26**, 341), the sulphur being absorbed as sulphuric acid in water or alkali. Hydrogen peroxide is added to the solution to ensure conversion of any sulphur dioxide to sulphuric acid, which is then precipitated with barium chloride solution. The results showed satisfactory agreement.

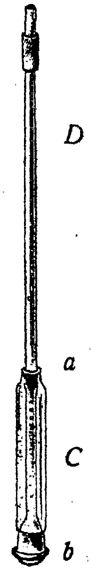
O. E. M.

Estimation of Sulphuric Acid. K. K. Järvinen. (*Ann. acad. scient. Fennicæ*, 1911, Serie A.2. Nr. 16; through *Chem. Zentralbl.*, 1912, I., 526.)—By the method given, about ·0025 grm.-molecule of sulphuric acid is titrated with a maximum accuracy of 0·2 per cent. The following ions may be present: sodium, potassium (magnesium), ammonium, in almost unlimited quantity; calcium, aluminium, manganese, zinc, iron, to the extent of 4 equivalents; copper, 1; chloride, 8; nitrate and borate, 4; phosphate, 2; oxalate, 1. Strontium, lead, chromium, and chromate ions must be absent. The solution, containing ·002 to ·003 grm.-molecule sulphate in 40 to 50 c.c., is neutralised if necessary until about 1 to 3 equivalents (= 2·5 to 7·5 c.c. 2 N) hydrochloric acid are left free; 3 equivalents are only required if 2 of phosphoric acid or 1 of oxalic acid are present. Ferric salts, 1 to 4 equivalents, are reduced by boiling with ½ to 2 equivalents of hydroxylamine. The solution is boiled up and slowly precipitated with about 40 c.c. (1 to 5 equivalents) of hot benzidine hydrochloride solution ($\frac{N}{5}+\frac{N}{10}$ HCl). The liquid is now evaporated to one-half, cooled, filtered, the precipitate washed with water containing benzidine sulphate, transferred to the original flask, and titrated hot with $\frac{N}{5}$ sodium hydroxide free from carbon dioxide, and phenolphthaleïn. The result is usually not more than 0·1 to 0·2 per cent. high. O. E. M.

Rapid Method of Estimating Zinc. K. Voigt. (*Zeitsch. angew. Chem.*, 1912, **25**, 205-206.)—V. Hassreidter has stated (*Zeitsch. angew. Chem.*, 1911, **24**, 2471) that the author's method (Analyst, 1912, 35) is open to objection on the ground that zinc may be underestimated owing to the re-formation of zinc silicate in presence of hydrated silica. The author points to the test numbers in his earlier paper for proof of the accuracy of the method, and adds that many of those test numbers were obtained in presence of 30 to 40 times as much "soluble" silica as zinc. He also points out that Hassreidter's experiments were not conducted in accordance with his directions, since Hassreidter used the smallest possible excess of ammonia, as he wished finally to complete the analysis by titration with sodium sulphide, which is rendered difficult or impossible by excess of ammonia. The large excess of ammonia directed to be used by the author prevents the zinc passing into the precipitate as silicate. A still larger excess is without influence on the results, but serves no useful purpose (*cf.* Analyst, 1912, 35). G. C. J.

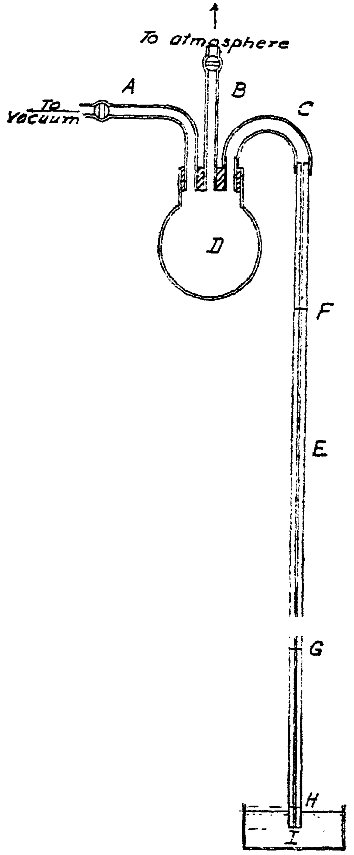
APPARATUS, ETC.

Automatic Pressure Pipette. W. Skinder. (*Chem. Zeit.*, 1912, **30**, 166).—The apparatus shown in the illustration is useful in cases where it is desired to bring two solutions to the same temperature before they are allowed to react with one another. It consists of a hollow rod, *D*, and a mantle, *C*, which are joined together by means of two ground-in joints. A small opening is provided at *a*, and another at *b*, which passes through both the hollow rod and the mantle. The apparatus is filled by raising the rod and immersing the mantle in the fluid; on withdrawing the apparatus from the fluid, it closes automatically. The hollow rod is then rinsed out with water, and the apparatus is placed in a vessel containing another fluid, and when the desired temperature is attained the contents of the apparatus are discharged by means of a pressure-bulb fitted to the upper end of *D*, the pressure produced raising the mantle, and allowing its contents to mix with the other fluid. W. P. S.



Device for Preventing the Bumping of Boiling Liquids. E. Pieszczek. (*Chem. Zeit.*, 1912, **36**, 198.)—A glass tube, 6 to 8 cm. long and 3 to 5 mm. in diameter, is drawn to a point at one end, into which is then fused a length of platinum wire to serve as a handle. The tube is immersed with its open end downwards, in a nearly vertical position, in the liquid. On heating the latter, bubbles of enclosed air escape from the tube, and all bumping is prevented, even in the case of liquids containing barium sulphate, or those covered with a layer of fatty acids. The air is never completely expelled from the small tube. C. A. M.

Method of Measuring Absolute Viscosity. H. P. Gurney. (*J. Amer. Chem. Soc.*, 1912, **34**, 24-28.)—The desirability of expressing the viscosity of liquids in absolute units is urged, and the apparatus figured is suggested for the purpose, for which the author claims extreme accuracy, simple and rapid manipulation, ease of working, shortness of time required for calibration, and the capacity for being used with opaque liquids and very small volumes. *F*, *H* is a capillary tube of suitable dimensions, graduated at *F*, *G*, and *H*, fixed vertically, and jacketed if necessary. The liquid to be tested is poured into *I* until it rises in the capillary to *H*, *B* being open to the atmosphere. *B* being then closed, *A* is opened, and the liquid drawn up above *F*. Atmospheric pressure is then restored, and the time required by the liquid to fall from *F* to *G* is taken by means of a stop-watch. Increased accuracy is attainable by repeating and averaging results. The density of the liquid may be obtained by any suitable method, and the radius of the capillary from the length and weight of a thread of mercury. A mathematical discussion of the results obtainable follows, from which the author deduces a very simple expression for the calculation of the viscosity. Thus, for a capillary tube of 1 metre in length, and bore of radius 0·03475 cm., of which the distance from *F* to *G* is 57·08 cm., and from *G* to *I* 21·37 cm.,



$$\mu = 0{\cdot}00247\gamma T,$$

where γ = the density of the liquid, and T = the time of flow from *F* to *G* in seconds, the radius of the dish *I* being 4·4 cm. H. F. E. H.

"Luminator" Treatment of Water. H. B. Lake. (*J. Soc. Chem. Ind.*, 1912, **31**, 57.)—It has been claimed (*J. Soc. Chem. Ind.*, 1910, **30**, 933) that the passage of a hard water over aluminium plates reduces its temporary hardness, and causes the deposit of its mineral matter, on boiling, as a sludge and not as a scale; the apparatus should be arranged north and south, exposed to direct light, and be given a weekly rest. The author has submitted water passed through either an aluminium chute or an aluminium tube, under the conditions stated, to soap-tests for total and permanent hardness, and to mineral analysis, comparing the residues obtained by evaporation of treated and untreated water. The differences were small. With one of the two waters treated there appeared to be a tendency towards the conversion of calcium salts from permanent to temporary hardness; with the other this was reversed. The treatment was without effect on the conversion of carbonates of lime and magnesia to sulphates, in hard water, by aluminium sulphate. O. E. M.

GOVERNMENT REPORT.

Report to the Local Government Board on Analyses and Methods of Detection of Certain Proprietary Substances sold as Preservatives for Milk, Cream, etc. G. Monier-Williams. (*Food Reports*, 1912, No. **17**, 1-7.)—A new preservative, sold under the name of "mystin," for preserving milk and cream, has recently been advertised as possessing the advantage that its presence cannot be detected by analysis. An analysis of the preparation showed that it had the following composition: Sodium nitrite, 9·85; formaldehyde, 0·30; water, 89·85 per cent.; the smell of the preparation indicated the presence of some essential oil, probably peppermint oil. The directions given prescribe the addition of 3 ounces of the preparation to 17 imperial gallons of milk. It was found that milk treated with this proportion of "mystin" failed to give the Hehner reaction for formaldehyde. This action of the nitrite in inhibiting the reaction appears to be due to the action of the nitrous acid with the tryptophane which is present among the products of hydrolysis of casein, rendering the tryptophane incapable of condensing with formaldehyde. If, however, the nitrous acid in the acidified milk be destroyed by some such substance as urea, the milk treated with "mystin" readily yields a coloration with Hehner's test. Five c.c. of the milk should be treated with 0·05 grm. of urea and 1 c.c. of $\frac{N}{1}$ sulphuric acid, the mixture heated for two minutes in a boiling water-bath, then cooled, and tested in the usual way. The quantity of formaldehyde may be estimated by the method described by Shrewsbury and Knapp (ANALYST, 1909, **34**, 12), after the milk has been treated with urea. In view of the possibility that "mystin" may have already come into extensive use among milk vendors, it would appear to be advisable to include the Griess-Ilosvay test for nitrites among the routine tests applied to milk and cream samples; milk containing "mystin" gives a pink coloration almost immediately with this test, and if a positive reaction is obtained, formaldehyde should be looked for by the method given above. It is pointed out that a quart of milk (treated with the proportion of "mystin" directed) would contain 2 grains of sodium nitrite, which is the maximum pharmacopœial dose of this salt. Sodium nitrite is a dangerous drug, with a powerful action on the heart, and the risk of using any preparation containing a comparatively large amount of sodium nitrite is universally recognised.

Reference is made to certain other preservatives for milk, cream, and margarine, to which the attention of the Board has recently been drawn. One of these, sold under the name of "accoine," consisted of an aqueous solution containing 13·97 per cent. of sodium benzoate, and 1·94 per cent. of sodium carbonate. It is claimed also for this preparation that it cannot be detected by analysis, but the presence of benzoic acid in milk and cream may be detected by the test described by Richmond (ANALYST, 1907, **32**, 151). Another substance, sold as a preservative for margarine, was found to consist almost entirely of sodium fluoride, whilst a cream preservative examined consisted of a mixture of sodium meta-bisulphite and sodium sulphate.

W. P. S.

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REVIEWS.

FOOD AND DRUGS. VOL. I.: THE ANALYSIS OF FOOD AND DRUGS. By E. J. PARRY, B.Sc. (Lond.), F.I.C. 1911. Pp. 715. London: Scott, Greenwood and Son. Price 21s. net.

The number of English textbooks on the analysis of foods and drugs is so limited that the appearance of a fresh claimant for space on our shelves need not, perhaps, excite surprise. The time has come, however, when single works covering such a wide range cannot be written by one individual from his own personal experience and knowledge, and the place of such encyclopædic enterprises as this must soon be taken by monographs on special subjects, or groups of subjects, by those who have made them their life's study.

The volume before us necessarily resembles in many points its predecessors, but, without any increase in bulk, it probably exceeds in range any of its rivals, owing chiefly to the omission of matters of historical and theoretical interest and of descriptions of apparatus, and, perhaps still more, to the inclusion of only "live" methods of analysis.

The book is written with a strict eye to utility, and if for the sake of brevity and convenience the style has suffered, something may be forgiven.

It is divided into two parts containing twelve chapters. Part I. treats of Food and Drinks, beginning with Tea, Coffee, and Cocoa, and ending with a chapter by Professor Greenish, on what is somewhat unhappily called "Microscopical Analysis," in which the apparatus, reagents, and general methods for preparing substances for microscopical examination are dealt with shortly and clearly. All the ordinary foods are referred to, and, in addition, some articles about which most of the general textbooks have little or nothing to say—*e.g.*, Macaroni and Italian pastes, meat extracts, and flesh foods, with a section on the properties of ptomaines, and the methods employed for their detection.

Part II. is devoted to Drugs, and is in some ways the most valuable part of the book, inasmuch as a great deal of information, which has hitherto only been available in isolated papers scattered through various journals, is here brought together for the first time. In addition to this, much of the ground covered comes within what may be considered as Mr. Parry's own special province, on which he is able to give authoritative and first-hand information. In the five chapters into which it is divided, the author adopts a system of classification which is novel, and though not without advantages is not, in our opinion, very logical or convenient, as it herds together the most unlike substances, and at the same time separates the preparations from the substances from which they are made. The first of these chapters is a perfect Noah's ark, for in it we find crude drugs—other than those containing active principles capable of estimation—gums, balsams, and resins cheek by jowl with roots, such as liquorice and male fern, animal derivatives such as musk and pepsin, organic preparations such as spirit of nitrous ether and the tinctures, liquors, and spirits of he Pharmacopœia, some of the substances from which these latter are derived being found in this and some in the next chapter.

Subsequent chapters deal with "Drugs containing Alkaloids, etc.," "The Essential Oils of the British Pharmacopœia," "The Fixed Oils, Fats, and Waxes of the British Pharmacopœia," "The Chemicals of the Pharmacopœia." In the case of the latter more than one-third of the space is taken up with the tests for arsenic and lead. The only chemicals treated at any length are saccharin, salicylic acid, camphor, and chloroform, the rest being dealt with in tabular form, the melting-point, boiling-point, solubility, and limits for arsenic and lead, being usually given, with a statement, in some cases, of the likely adulterants. Practically, the tables are an abstract of the British Pharmacopœia descriptions and requirements, with occasional criticisms. The most valuable portions of the second part of the book are the chapters on the drugs containing alkaloids and on the essential oils, which are commendably full and embody much of the author's personal experience.

While the work as a whole may be highly commended, it suffers considerably from haste in production and insufficient revision. The wording is, as a rule, clear and concise, but in the matter of style leaves a good deal to be desired.

In the case of the chapters dealing with foods, uniformity of treatment, especially as regards the headings of the paragraphs, might have been carried a good deal further, and a more systematic division of the chapters into sections and subsections, with appropriate headlines would have made for ease of reference, and would have prevented the author from omitting some by no means unimportant matters. The articles on individual foods are certainly broken up into paragraphs with descriptive headings, but the plan adopted differs in the case of almost all of them—for instance, under cheese we find "the adulteration of cheese," under tea "the adulterants of tea," but under butter and milk no corresponding headline appears, although there is "calculation of adulteration" in the case of milk. The difficult question of food standards is not very satisfactorily dealt with, for the word in this sense does not occur in the index, and although well-selected tables of analytical data are given for the articles dealt with, and definite suggestions are frequently made as to the basis on which to calculate the percentage of adulteration, it is necessary in many cases to read the whole article through in order to find it. In other parts of the book the word "standard" is somewhat loosely used, as in one place it is applied to the definitions and descriptions of substances given in the British Pharmacopœia, while in another, a table showing the range of variation of the analytical constants of the British Pharmacopœia tinctures is headed "Standards for Tinctures," etc. A more important point is the omission to refer, except in one or two instances, to the numerous standards and definitions of articles of food drawn up by the United States Department of Agriculture, which, though not binding in this country and by no means universally accepted here, nevertheless carry a good deal of weight. Certain other omissions should also be mentioned here. No reference is made to the reports issued by the Local Government Board on Vinegar (1908), on Baking Powders, and on the Bleaching of Flour (1911). (As these were issued about a year ago there is some excuse for them not appearing, although the book as a whole has been brought very well up to date.) The Report of the Departmental Committee on Preservatives and Colouring matters only receives a passing mention. In connection with the analytical methods, there are a few similar omissions of processes to which reference might reasonably be expected, but they must be regarded, in the case of a book of such wide range, rather as departures from the ideal than as serious blemishes.

There are also a number of printer's errors which a more careful revision would have avoided. Fortunately, most of them do not alter the sense—*e.g.*, "Hanwill" for "Hamill," "Avi-Lallement" for "Ave-Lallement," "Badouin" for "Baudouin," "*Capsicum fastigatum*" and "*C. putescens*" instead of "*fastigiatum*" and "*frutescens.*" More unfortunate is the reference to the rotation of "1 mg. of quartz." The definition of specific rotation has also suffered in some way at the printer's hands—in fact, the whole of the section dealing with the Polarimeter is hardly up to the author's usual standard of lucidity.

Speaking generally, and it is impossible to criticise in detail a book which is almost an encyclopædia, the work of compilation has been excellently done, the selection of methods and of tables of analysis—always a difficult task—has been most judicious, very few obsolete processes are included, and the best, as well as the newest, work is referred to. The microscopical examination of the vegetable foods and drugs is dealt with briefly but clearly, and in a really useful manner; the illustrations are clear, and will be as helpful as illustrations in the absence of actual specimens are ever likely to be. Mr. Parry is to be congratulated on having so satisfactorily accomplished such a colossal task. C. H. CRIBB.

FOOD AND DRUGS. By E. J. PARRY, B.Sc. (Lond.), F.I.C. Vol. ii.: The Sale of Food and Drugs Acts, 1875-1907. Pp. 181. London: Scott, Greenwood and Son. 1911. Price 7s. 6d. net.

In view of the existence of other recently published works, Mr. Parry's task in dealing with the Sale of Food and Drugs Acts from a legal standpoint cannot have been easy. But he has wisely endeavoured to break fresh ground by giving the reader the benefit of his personal views on many points, and several of his suggestions and submissions have both originality and force. Especially good are Mr. Parry's observations on the British Pharmacopœia in relation to the Sale of Food and Drugs Acts. For the purpose of this discussion the drugs of the B.P. are classified in five groups, and the effect of High Court decisions in such cases as Hudson *v.* Bridge is stated with admirable clearness and accuracy. Cogent arguments are adduced in support of the contention that convictions under the Sale of Food and Drugs Acts for the sale of distilled water containing traces of impurities are wrong, on the ground that distilled water is not a drug. Mr. Parry thinks that any conviction for the sale of golden syrup containing glucose would be upheld by the High Court; that there need be no soda in a "whisky and soda" sold at a refreshment bar; and that if a sample of pills, tablets, or capsules, be taken by an inspector under the Sale of Food and Drugs Acts, it is necessary, under the decisions in the cases of Mason *v.* Cowdary and Smith *v.* Savage, that each pill, tablet, or capsule be divided into three parts. These and other personal opinions are always interesting, and, even if erroneous, add to the value of the work. The ordinary lawyer is too cautious thus to volunteer his views.

It is interesting to note that Mr. Parry is among those who hold that the Public Health (Regulations as to Food) Act, 1907, clothes the Local Government Board with wide powers to make binding regulations and "standards" with respect to articles of food, to prohibit preservatives, and so forth. Over eight pages in the body of the work are occupied by the text of the regulations of the Institute of Chemistry for the analytical qualification.

The author makes a good point, which is not perhaps universally understood—namely, that a private purchaser desiring to act as a prosecutor under the Sale of Food and Drugs Acts can rely on the evidence of any analyst, and need not necessarily make use of the power given him by Section 12 of the Act of 1875 to have his sample analysed by the public analyst for the district. Mr. Parry includes in this book six specimen certificates, against which he suggests no technical objection could be sustained. This is probably true; but the specimen certificate for diluted spirits is needlessly involved, and would convey singularly little meaning to an ordinary bench of magistrates. Much better, surely, is the form of certificate adopted by several well-known public analysts as follows:

". . . . . . parts as under:

Whisky, 25 degrees under proof … …	78
Added water … … … … …	22
	100

"This opinion is based on the fact that the sample was 41·5 degrees under proof. To whisky of 25 degrees under proof, the above-mentioned proportion of water must be added to reduce it to 41·5 degrees under proof. Genuine whisky should not be more than 25 degrees under proof."

Mr. Parry states that any person desiring to appeal to the High Court by way of special case "must apply for a case to be stated within three days of the Justice's decision." This is not so. He must apply within seven days, and the case must be stated within three months. And surely some sort of information ought to have been given as to the formal steps which have to be taken in lodging such an appeal. Another unsatisfactory statement is that where proceedings are taken under the Merchandise Marks Act "it is sometimes necessary to institute proceedings in the High Court, as in the case of limited companies." Is it ever necessary to institute proceedings in the High Court except in the case of a limited company refusing to be dealt with summarily?

The index is a little scanty, and the editing of the work slovenly in the extreme. *Res ipse loquitur* sets the teeth on edge. The date of the last edition of the British Pharmacopœia is twice given as 1908. The case of Parker *v.* Alder, reported in Law Reports, 1899 (1 Q. B., 20) is twice cited as Parker *v.* Adler (1 Q. B., 20) 1898. The case of Fortune *v.* Hanson is quoted at p. 44, but the only reference there given is "(1 Q. B., 202)." Such a reference is of course utterly valueless without a reference to the year in which the report appeared. At p. 5 the year comes first in each reference to volumes of the Law Reports; in the whole of the rest of the book this proper and sensible practice does not obtain. Mr. Parry has made remarkably little use of the ordinary typographical resources which can be made of such assistance in legal textbooks—a fact which detracts enormously from the practical value of the work. There are few subheadings and little variation in the fount of type used. Thus, at p. 79, under Section 18 of the Sale of Food and Drugs Act, 1875, is set out the schedule giving the form for the analyst's certificate. This is printed in exactly the same way as Mr. Parry's comments (which follow it), and there is nothing to indicate where the schedule ends. Indeed, no one who was not already familiar with the schedule would know whether the last sentence in the schedule is statutory or whether it is an observation on the part of Mr. Parry. In one instance, however, there is too much variation. The prefatory words to Acts of Parliament (Be it enacted . . . etc.) are printed sometimes in ordinary type and sometimes in heavy black type, without any apparent reason for the distinction. Many proper names are misspelt, Macfadden, Londsdale, Meddlings, and Hillard being examples. There is an irritating absence of uniformity in the references to the reports of the Inspectors of Food to the Local Government Board, and at p. 11, dealing with the same subject, Mr. Parry writes of "these recommendations by the Local Government Board *committees*." The Pharmacy Act of 1868 is wrongly cited as the Sale of Poisons and Pharmacy Act, 1868. In the most recent edition of another text-book this unfortunate statute was cited as the Sale of Provisions and Pharmacy Act, 1868.

On the whole it may be said that while this second volume contains much that is sound, original, and interesting, it bears evidence of careless preparation, and is hardly likely to be regarded by those who have to conduct cases under the Sale of Food and Drugs Acts, as a book to be taken into the Police Court as a matter of course.

R. A. ROBINSON.

MILK AND THE PUBLIC HEALTH. W. G. SAVAGE, B.Sc., M.D. Macmillan and Co., Ltd., London. 1912. Pp. xviii + 459. Price 10s. net.

The author in his preface states that this volume is entirely concerned with milk in its relationship to the general health of the community. In Part I. he makes an endeavour to give a summary of our scientific knowledge, and of the facts with which the patient labours of many investigators have endowed us, as to the bacterial contamination of milk and its relationship to disease; in Part II. the practical bacteriological examination of milk is described in some detail, while in Part III. the administrative side of the subject is dealt with.

It is recognised that our present knowledge of the subject is far from complete, and though great strides have been made within a very few years, the chief fault of the book is the failure to grasp the fact that great advances in the technical handling of milk have also taken place in recent years; for instance, the question of preservatives is discussed quite fully, and tests are given for the leading substances that have been used; yet, although he has been obliged to go back to 1899 for any statistics as to their prevalence, the author has not realised that their use in milk has practically ceased, owing to the realisation by the technical man of the fact that it is better, and even cheaper, to keep milk by refrigerating than by drugs. The question of refrigeration is dismissed in less than a page, and no mention whatever is made of the small refrigerating plants designed specally for use with milk which are now so generally employed; there are also other reforms taking place from within the dairy industry —such as the extending use of an efficient pattern of milk churn and the provision of washing accommodation—which the author ignores.

Apart from the want of technical knowledge, and an equally excusable weakness in some of the chemical portions, the author has presented an excellent *résumé* of our present knowledge. The practical bacteriological section is less satisfactory, not through any shortcoming of the author, except possibly that he is too anxious to give all the information, and is, therefore, not sufficiently dogmatic. This will best be illustrated by a criticism of the detection of *B. coli* and allied organisms; the method is, broadly, MacConkey's, as the milk is added to lactose bile salt broth, and if acid and gas fermentation is produced, the solution is plated out on lactose bile salt neutral red agar (not MacConkey's most recent formula), which is preferred to the other media which have been proposed. What the characters of *B. coli* are is not strictly defined, though it would appear that the author considers as this all organisms which grow as red colonies on the bile salt lactose neutral red agar, and which do not liquefy gelatine, which give acid and clot in milk, ferment lactose, and which produce indol; whether even the indol production is essential is doubtful, as it is stated that the relative significance of the strains that produce indol, and those which do not, has yet to be accurately determined. It is true that the differentiation of the various species by the fermentation of sugars is discussed very fully, but the final words of this discussion are "the practical value of these tests has yet to be demonstrated."

It would have been far better, from a practical point of view, had the author selected a particular technique, described it in detail, and said, autocratically, the particular organisms identified by this method may be considered for practical purposes as *B. coli*; it is important to describe the method fully, as modifications in detail often produce large differences in the result; thus, while it is mentioned that better tests for indol are now available, no mention whatever of the *p*-methylaminobenzaldehyde method is given, though this is very much more sensitive and reliable than the old nitrite method.

In conclusion, this book is written by a bacteriologist for bacteriologists, and for the trained man who can use it with discrimination the work is full of valuable information; but the analyst whose knowledge of bacteriology is not extensive had better leave it alone.

H. DROOP RICHMOND.

CHAUX, CIMENTS, PLÂTRES. By E. LEDUC and G. CHENU. (Manuels Pratiques d'Analyses Chimiques), Paris and Liège. Ch. Béranger. 1912. Price 6 francs.

This little volume forms one of a series designed for the use of public analysts, and deals with the chemical, physical, and mechanical methods of controlling the quality of lime, cement, and plaster. The analytical methods described are almost all familiar, and little notice is taken of recent improvements in cement analysis, but the directions are clearly given and would in most cases lead to satisfactory results. Exception may, however, be taken to certain points. It is unfortunate that the misleading sugar test for "free lime" should be retained, it having long been known that the proportion of lime extracted from a cement by means of a sugar solution has no practical or theoretical significance. The methods proposed for the detection of blast-furnace slag in Portland cement are also unsatisfactory, the separation by means of methylene iodide being only applicable to coarsely-ground cements, and failing almost completely with the modern very finely ground product. Fresenius' method, based on the difference of reducing power towards permanganate, is not mentioned. Some confusion may be caused by the use in one place of Michaelis' "hydraulic modulus," and in another of a "hydraulic index" which is the reciprocal of the former, and this confusion is increased by "inférieur" being printed instead of "supérieur" at the foot of p. 95. The methods of making the usual tests of fineness, constancy of volume, tensile strength, permeability, etc., are clearly described and illustrated, the authors generally following Le Chatelier, and there are some useful tables of results obtained from cements of French manufacture. The appendix contains a full account of the French specifications for various classes of material, as well as those relating to Portland cement laid down by the Governments of Prussia, Egypt, Argentine, and Switzerland, and the American Society for Testing Materials, but the British Standard Specification is not included.

The book should be useful to French public analysts, and to others who have frequent occasion to examine products of French manufacture. C. H. Desch.

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INSTITUTE OF CHEMISTRY.

The Thirty-fourth Annual General Meeting of the Institute of Chemistry was held on March 1 at 30, Bloomsbury Square, Dr. George Beilby, F.R.S., the President, occupying the chair.

After the routine business had been transacted, the Retiring President delivered his address. He showed that, during his period of office (1909-1912), the Institute had increased by 102 members and 86 students, while the number of candidates for examination during the same period had increased by about twenty. The scheme for raising a fund for new buildings for the Institute had made substantial progress, and had reached the sum of over £8,500 of the £15,000 considered necessary for the erection of a simple but dignified building suitable for carrying on the work of the Institute. Speaking of the prospects of the profession of chemistry, he believed that there was plenty of room for men of the right stamp, both in educational and industrial work. The Institute had been able to secure appointments for many young members of the profession, and, indeed, lately it had been difficult to find candidates for appointments offering quite good commencing salaries and fair prospects. Touching on the difficulties which confront public analysts and private practitioners, he referred to the attempts made on the part of certain local authorities to lower the status of the professional chemist by offering appointments at ridiculous remuneration. The Institute had decided to take a new and somewhat drastic step by issuing a notice (ANALYST, 1912, 119) to Fellows and Associates, advising them not to apply for appointments advertised by the Metropolitan boroughs of Lambeth and Wandsworth, both of which at the present time seek to appoint a public analyst who will be required to provide a laboratory, duly equipped, and a deputy, and to examine possibly as many as 2,000 samples, for £500 per annum; while the County of Antrim seek to appoint a public analyst to examine an indefinite number of samples—almost certainly over 800 a year—at a salary of £150. The more enlightened municipal bodies realise that the proper administration of important Statutes, such as the Sale of Food and Drugs Act, cannot be expected unless they succeed in attracting to their appointments men of competence and integrity, and, moreover, men who can hold their own as responsible representative officers of their authorities. The Act is as much a Statute against fraud as in the interests of public health, and it must be clearly understood that the public analyst is in no way subject to the control of the Medical Officer of Health. It was a false notion that medical men were capable of controlling chemical laboratories. Very few of them possessed anything approaching a competent knowledge of chemistry in any one of its branches, and it was astonishing to find Government authorities in some of our overseas dominions requiring doctors of medicine to supervise qualified professional chemists engaged in laboratories mainly devoted to the analysis of metals and ores. DR. BEILBY referred to important changes which were being made in educational appointments, particularly the Chairs of Chemistry at the Royal College of Science, Dublin, Oxford University, and University College, London, indicating how men who had achieved great things were making way for the younger generation of chemists, to whom we look to maintain the honour of the British school of chemistry, to which their predecessors had contributed so much. During the past year the Council had inaugurated a new scheme of lectures, mainly intended to help registered students and the younger members, such lectures being given by acknowledged experts in various branches, and illustrating the actual work of practice as distinct from purely academic training.

A ballot having been taken, Dr. George Beilby, F.R.S., Professor Percy F. Frankland, F.R.S., Mr. David Howard, and Sir William Tilden, F.R.S., were declared elected as censors.

The following were elected officers and Council:

President.—Raphael Meldola, D.Sc., LL.D., F.R.S.

Vice-Presidents.—George Thomas Beilby, LL.D., F.R.S.; Frank Clowes, D.Sc.; George McGowan, Ph.D.; Sir Alexander Pedler, C.I.E., F.R.S.; John Millar Thomson, LL.D., F.R.S.; Sir William Augustus Tilden, D.Sc., LL.D., F.R.S.

Hon. Treasurer.—Alfred Gordon Salamon, A.R.S.M.

Members of Council.—Francis William Frederick Arnaud; Edward John Bevan; Bertram Blount; Arthur George Bloxam; James Connah, B.Sc.; William Salvador Curphey; Cyril Dickinson, B.Sc.; James Johnston Dobbie, Ph.D., LL.D., F.R.S.;

William Porter Dreaper; Martin Onslow Forster, D.Sc., F.R.S.; John Alfred Foster; Sir Richard Garton; Frank William Harbord, A.R.S.M.; Otto Hehner; Gordon Wickham Monier-Williams, B.A., Ph.D.; William Henry Perkin, LL.D., Ph.D., F.R.S.; Thomas Slater Price, D.Sc., Ph.D.; Charles Proctor; Sir Boverton Redwood, Bart., D.Sc.; Henry Droop Richmond; Clarence Arthur Seyler, B.Sc.; Alfred Smetham; Thomas Stenhouse, jun., B.Sc., A.R.S.M.; Oliver Trigger; Edward William Voelcker, A.R.S.M.; John White; Sydney Young, D.Sc., F.R.S.

MAY, 1912. Vol. XXXVII., No. 434.

THE ANALYST.

OBITUARY.

ARTHUR EDWARD EKINS, J.P., F.I.C.

Arthur Edward Ekins, whose death on March 12 was briefly recorded in our last number, was one of the early members of the Society of Public Analysts. He was the son of a farmer in the Fen country, was born in the Isle of Ely in 1852, and educated at St. Ives Grammar School and at Melbourn, Cambridge. He was originally trained for pharmacy, and in 1876 established a pharmaceutical business at St. Albans. In conjunction with this he carried on an analytical practice, chiefly in connection with agricultural matters, as Analyst to the Herts Agricultural Society. On the death of Charles Heisch, the County Analyst, in 1892, Mr. Ekins was appointed Public Analyst for the County under the Sale of Food and Drugs Act, shortly after which he retired from his pharmacy in order to devote himself more fully to his professional duties, as well as to the municipal work of the Corporation of St. Albans, in which he had always taken an active part. He occupied the position of Mayor of the City in the year of King Edward's Coronation, and it is recorded that during his mayoralty he attended in his official capacity no fewer than 600 meetings.

In addition to working constantly on the various committees of the Corporation (including the Library Committee, the Assessment Committee, and the Pensions Committee), he laboured assiduously as a Magistrate, Poor Law Guardian, Overseer, Churchwarden, and Manager and Treasurer of the local Christ Church School. He also held the position of Chairman of the local County Council School Managers, of the Joint Committee of the Sisters' Hospital and of the St. Albans Building Society. He took a prominent part in the masonic life of the county, in which he held honours as a Provincial Grand Officer.

All these forms of civic and social activity left him but little time for active contribution to the advancement of chemical knowledge by original work, but he did his work as County Analyst conscientiously and effectively, his duties extending to the annual examination of a considerable number of water samples and sewage farm effluents, in addition to his routine work under the Sale of Food and Drugs Act. It was very rarely that Mr. Ekins missed his attendance at the monthly meetings of the Society of Public Analysts or at the social gatherings connected with them. He served the Society not only as a member of Council, but as Vice-President, in which capacities his assiduous attendance at both Council and Committee meetings was rendered the more useful by reason of the sound judgment and business ability which he always brought to bear on the matters under discussion. He had, moreover, a high sense of professional honour, which gained for him the respect and esteem of his colleagues, while his geniality and kindheartedness equally won their personal affection.

BERNARD DYER.

JOHN PATTINSON, J.P., F.I.C.

JOHN PATTINSON was born at Alston, in Cumberland, on February 13, 1828. His schooldays were spent in his native town, and, as a youth, he was brought to Newcastle by his distant relative Hugh Lee Pattinson, well known as the inventor of the Pattinson process for desilverising lead. Hugh Lee Pattinson was a proprietor of the Felling Chemical Works, and there John Pattinson received his early chemical training. He remained there for ten years, and then went to Middlesbrough, under another north-country leader of industry, Isaac Lowthian Bell. He thus obtained a first-hand knowledge of the processes involved in the alkali manufacture, and in the iron and steel manufacture of the time. In 1858 he returned to Newcastle, and took over the practice of analytical and consulting chemist of William Crowder, in the Side, out of which the present firm of J. and H. S. Pattinson has developed. In 1868, along with other devotees of chemical science in the town and district, he took part in the foundation of the Newcastle Chemical Society; he was treasurer of that Society until the date of its absorption into the Society of Chemical Industry in 1882, president in 1874-5, and contributed to it papers on the determination of peroxide of manganese, on the quality of the small coal used on the Tyne, on the determination of arsenic in copper, and on other subjects. When the British Association met in Newcastle in 1863, he read a paper on the pyrites used on the Tyne. His practice dealt largely with iron and manganese ores, and in 1879 he read a paper (*J. Chem. Soc.*, 1879, 365) on the determination of manganese, describing a volumetric method which, with slight modifications (described in *J. Soc. Chem. Ind.*, 1891, **10**, 333), is still widely used, and is probably the most accurate method known. Several other papers and notes appear under his name, alone, or in association with his son the late Dr. H. S. Pattinson or with Dr. Dunn, in the Society of Chemical Industry's *Journal*. He was one of the earliest of Public Analysts; he was appointed Gas and Water Examiner for Newcastle in 1863 or earlier, Food Analyst in 1871, and later became, under the Food and Drugs Acts, Public Analyst for the town, for the county of Northumberland, and for other boroughs in the district. He had been a Vice-President of the Chemical Society, and Vice-President of this Society; was an original Fellow of the Institute of Chemistry, which he had served as member of Council and as Censor; and an original member of the Iron and Steel Institute. He was an enthusiastic musician, was a Vice-President of the Newcastle Literary and Philosophical Society, a justice of the peace, and greatly interested in social and philanthropic matters. He kept up all his interests, professional and other, to the last; and after only a few days' illness he died on March 28 last.

J. T. DUNN.

EDWARD DIVERS, M.D., F.I.C., F.R.S.

EDWARD DIVERS was born in London in 1837, and was educated at the City of London School. He subsequently studied chemistry under Professor A. W. Hofmann at the Old College of Chemistry, Oxford Street, and in 1866 he graduated as M.D., at Queen's College, Galway. He was in private practice until 1873, when, at the invitation of the Japanese Government, he, with other Englishmen, went to that country to establish a College of Engineering. This College ultimately became part of the Imperial University, and Divers was appointed Professor of Chemistry. In 1886 he retired, and was made Emeritus Professor; he received the second class of the order of the "Sacred Mirror," and the third class of the order of the "Rising Sun."

On his return to England he at once took an active part in the affairs of the Chemical Society and the Society of Chemical Industry. In 1893 he was elected a Fellow of the Royal Society.

The name of Divers will always be associated with the subject of the oxides of nitrogen and sulphur, and with hyponitrites, of which he was the discoverer. In conjunction with Shimose, he was the first to show that when tellurium monoxide is heated in hydrochloric acid gas, it is converted into tellurium dichloride.

Divers will be missed by a wide circle of friends, for his charming personality, kindliness, and urbane manners endeared him to many.

He died on April 8.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE Monthly Meeting of the Society was held on Wednesday evening, April 3, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous Ordinary Meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. L. Goodban, A.I.C., and R. F. Easton were read for the second time, and certificates in favour of Messrs. Harold Thomas Cranfield, Assistant Lecturer in Chemistry and Assistant Analyst at the Midland Agricultural and Dairy College, Kingston, Derby; Ernest Gabriel Jones, M.Sc. (Victoria and Liverpool), F.I.C., City Laboratories, 36, Dansie Street, Liverpool, Deputy Public Analyst and Deputy Agricultural Analyst for the City of Liverpool; and William Henry Roberts, M.Sc. (Victoria and Liverpool), F.I.C., City Laboratories, 36, Dansie Street, Liverpool, Public Analyst for the City of Liverpool, and the County Boroughs of Blackburn, Preston, Bootle, and Barrow-in-Furness, Official Agricultural Analyst for the City of Liverpool and Borough of Bootle, were read for the first time.

Messrs. M. E. Balston, M.A., H. A. Caulkin, B.Sc., F.I.C., and C. R. Wilkins, B.Sc., were elected members of the Society.

The following papers were read: "The Separation of Arsenic from Antimony," by Stanley W. Collins, B.Sc., F.I.C.; "The Estimation of Ferric Iron in Presence of Certain Organic Substances," by John Theodore Hewitt, M.A., D.Sc., Ph.D., F.R.S., and Gladys Ruby Mann; "The Relation of the Kirschner and Polenske Values in Margarines containing Cocoanut or Palm Kernel Oils," by E. Richards Bolton, H. Droop Richmond, F.I.C., and Cecil Revis; "Note on the Milk of a Small Herd," by Edward Russell, B.Sc., F.I.C.; and "A Convenient Apparatus for Obtaining an Average Sample of Gas and for Regulating the Flow of a Gas into an Evacuated Vessel," by Frank Sturdy Sinnatt.

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THE SOURING OF MILK.

By H. DROOP RICHMOND, F.I.C., and HORACE C. HUISH.

(*Read at the Meeting, February* 7, 1912.)

Part I.—Method used.

The rate of souring of milk is determined by a series of acidity estimations at intervals, and depends on the difference in the acidity at a given interval from that at the commencement; as milk is a substance in which the concentration of the hydrogen ions is low, the end reaction with the most convenient indicator—phenolphthaleïn—is not very sharp. We have taken the point of neutralisation to correspond with the appearance of a faint pink colour, but we find that under varying conditions of light, and to the eyes of different operators, there is an appreciable variation in the end-point; between two or more experienced operators who have learned to appreciate the point at which a very faint pink colour appears there may be a difference of as much as 2°, and this difference may increase sometimes up to 5° with operators of less experience. Even the same operator does not always make very concordant duplicate determinations if the conditions differ.

Even using a blank, we do not find that the accuracy of the determination is greatly increased, and we have experimented in several directions with a view of increasing the accuracy of this determination. By colouring the milk faintly green (the colour complementary to pink), it was found that the accuracy was slightly, though not very markedly, increased, especially if a green-tinted blank were used; but after a prolonged experience of this method we came to the conclusion that it was wrong in principle to try and work from a colour, but that the best method would be to endeavour to work to the appearance of a definite tint.

We find that, although the colour given by rosaniline to milk is not exactly the same tint as that yielded by phenolphthaleïn when made alkaline, it is in very dilute solution sufficiently near to enable it to be used as a standard tint, and the method we now employ is this: Eleven c.c. of the milk to be tested is placed in each of two similar beakers, and to one, which serves as the standard tint, one drop of a 0·01 per cent. solution of rosaniline acetate in 96 per cent. alcohol is added; to the other 1 c.c. of a 0·5 per cent. solution of phenolphthaleïn is added, and the milk is titrated with $\frac{N}{11}$ strontia solution till its tint is equal to that of the standard. The result expressed as cubic centimetres of normal alkali per litre we term "degrees of acidity" (rosaniline standard), or, more shortly, R. S. We find that not only do different operators obtain closely concordant results, but that the nature of the light makes no difference in the end-point. The results are about 1·5° higher on the average than those obtained when titrating to the appearance of a faint pink colour.

By the use of the rosaniline standard we have far more confidence in the accuracy of the determination of small increases in acidity, and we may mention incidentally that the accuracy of the determination of the aldehyde figure, which depends likewise on a difference between two titrations, has also been increased.

PART II.—THEORETICAL CONSIDERATIONS.

In endeavouring to trace the course of the souring of milk we have made certain assumptions. The first is that it is due to micro-organisms, or that, if due to enzymes secreted, these do not diffuse away from the micro-organisms into the liquid; or, in other words, the amount of acidity produced at any moment is proportional to the micro-organisms present at that moment. The second assumption is that the rate of diffusion of the acid produced away from the micro-organisms is rapid compared with the rate of production.

These assumptions lead us to the differential equation:

$$dm = \mathrm{k}da \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

(m = number of micro-organisms; a = acidity.)

If we make the further assumption that the increase in the number of micro-organisms is proportional to the number present at any moment, we may write from this assumption and from (1) the differential equation

$$da = \mathrm{k}'adt \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

(t = time),

which leads to the equation

$$\log a = \mathrm{k}'t + c \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (3),$$

which leads to a simple logarithmic curve for the souring of milk.

On the assumption that no increase of micro-organisms takes place, but that each micro-organism produces acid at a definite rate, the differential equation

$$da = \mathrm{k}''dt \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

results, leading to the equation

$$a = \mathrm{k}''t + c' \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (5),$$

which shows a straight line as the curve.

If we assume that the micro-organisms do not increase, and that the rate of production of acidity falls off in proportion to the amount of acid already developed acting for a unit of time, we arrive at the differential equation

$$da = k''' \frac{a}{t} dt \quad . \ . \ . \ . \ . \ . \quad (6),$$

which on integrating gives us—

$$\log a = k''' \log t + c'' \quad . \ . \ . \ . \quad (7).$$

The curve of this equation is a parabola, though not necessarily a rectangular parabola.

PART III.—EXPERIMENTAL.

From the mean of a very large number of acidity determinations at known periods of time we have constructed the table below. This shows the acidity developed at intervals of five hours from the time of milking, when the milk was kept at a temperature of 68° F. There has frequently been a difference of several units in the acidity developed in different samples, but by averaging the results of a large number we believe we have obtained a fair representation of the average souring of milk.

Average Souring of Milk.

Time in Hours.	Acidity.	Calc.	Time in Hours.	Acidity.	Calc.
10	0	0·2	60	71	77·1
15	0·1	0·5	65	74	74·3
20	0·3	1·1	70	76·5	77·1
25	2·3	2·5	80	82	81·5
30	6·0	5·7	90	85	85·0
35	13·0	13·1	100	88	88·9
40	30·0	29·9	110	91	90·7
45	50	50·5	120	93	93·1
50	62	61·1	130	95	95·2
55	67	66·8	140	97	97·0

The formula used for calculating the acidity up to 45° acidity is

$$\log_{10} a = 0{\cdot}072\, t - 1{\cdot}406,$$

which is of the type of (3); and that used after 45° is

$$\log_{10} a = 1{\cdot}621 + 0{\cdot}184 \log (t - 42{\cdot}2),$$

which is of the type of (7). It is interesting to note that these two curves do not meet, indicating that there is a short period during which another curve, possibly of the type of (5), expresses the results.

The change from one branch of the curve to another is quite sharp, and takes place at 45°, which is the point at which micro-organisms cease to develop in milk (*cf.* Revis and Payne, *J. Hygiene,* 1906, 216); there is no other break in the curve, that mentioned by one of us and Miller (ANALYST, 1907, **32,** 144) at 65° not having a real existence.

To try and elucidate this sudden change in the rate of souring of milk at the point when an acidity of 25° has been developed, we have made a number of careful experiments in which continuous observations were made over a period of forty-eight hours, determinations in some cases being made hourly. It will not be necessary to give these experiments in full. The following is an abstract of the results:

1. In three series of experiments in which milk to which a definite amount of alkali had been added, and milk to which a definite amount of acid had been added, were allowed to sour side by side, it was found that the point at which the break occurred was advanced or retarded by an amount equal to the quantity of acid or alkali added. Thus, a milk with an acidity of 18·2° was reduced in acidity to 3·3° by the addition of caustic soda, and increased to 28·6° by the addition of hydrochloric acid. The points at which the breaks occurred were 45°, 60°, and 34°, respectively.

2. It was found in every case that the addition of alkali slightly accelerated the rate of souring, and that of acid slightly retarded it, but to a very small extent.

3. Experiments on pasteurised milk to which either pure cultures of lactic organisms or a little stale milk was added showed a greatly increased rate of souring. The greatest acceleration of souring was shown by a sample of milk which was allowed to become partially sour, and was then neutralised with soda and pasteurised, and inoculated with a trace of the unpasteurised milk.

4. It was found that in every case, no matter what the rate of souring, the curves were very similar. There were, however, slight differences (best seen on plotting out the logarithms of the acidity against time). In some cases a distinct acceleration of the rate of souring was seen, and in others a very distinct retardation.

From these results we conclude that—

(*a*) On growing in milk the organisms developing acidity become more active.

(*b*) The organisms are retarded by the acidity they develop, and in the majority of cases this retardation is balanced by the acceleration due to their growing in milk.

(*c*) At an acidity equal to 45° the retardation becomes very great. The point 45° of acidity depends on the nature of the salts present in milk, as in whey it is about 30°, and in a lactose solution about 20°. It is probable that these acidities represent the same concentration of the hydrogen ions.

It follows as a corollary that the organisms which normally cause the souring of milk are not those whose normal habitat is milk, and it is not improbable that they are of intestinal origin.

DISCUSSION.

Mr. C. REVIS said that he was glad to hear that the authors did not consider that the ordinary lactic acid organisms found their natural habitat in milk. He (the speaker) had for some time been of opinion that the lactic acid organisms usually found in milk were derived principally from cow-dung. It might be contended that such organisms, when found in milk, often came from the udder, but *Streptococcus mastitis*, which grew in the udder in cases of mastitis, was probably from the same source. One observer, however, considered that lactic acid organisms were derived from the cow's saliva. He thought, however, that the cow-dung was the principal source of the organisms, and that milk was certainly not their natural habitat. The problem from a bacteriological point of view was a very difficult one, there being no means of ascertaining at any moment how many organisms were present in the milk, how many were active, or at what rate they were dying out. Grimme, in some work on this subject, had obtained curves identical with those obtained by the present authors, shewing that there was at first no measurable increase in acidity, although the organisms must have been multiplying. Was there a stage in the life of the organism which had to be reached before it exercised a certain activity, which afterwards gradually decreased? At the point at which there began to be a decrease in the rate of acid production, it might be that the action of the organisms was inhibited by the acid, but he thought it probable that a greater number of them were dying out at the same time, and that the decrease in acidity might be due simply to the fact that more of the organisms were caused to die off through the acidity they produced than were being produced by multiplication of those still active. He had found with *B. coli* that the activity of that organism and the results it produced were profoundly affected by slight changes in reaction of the nutrient medium. The lactic acid organisms were undoubtedly very sensitive to the acid they produced, for if a culture were left for a fortnight or three weeks it would become absolutely sterile. With a pure culture the problem was simpler, but if lactic acid organisms were grown in the presence of other organisms common to milk which did not themselves produce acid, the rate of acid production by the lactic acid organism was often much modified. It had to be remembered that up to the point of coagulation of the milk the organisms were not exposed to the same acidity as they were after that point. The lactic acid at first was neutralised by the calcium in combination with the casein, and although the casein exerted an equivalent chemical activity, it might not be equivalent bacteriologically, and so be less inimical to the development of the organisms.

Dr. Lessing mentioned that a method had been published by Dr. Schryver and himself some years ago for measuring bacterial reactions of this kind by means of electrical conductivity. They had found it possible, when the action of the bacteria was inhibited by antiseptics, to see whether the changes went on, and so to ascertain whether the action was due to enzymes alone, or to bacteria alone, or to both together.

The President asked whether the shape of the curve was not a good deal dependent on the conditions of the experiment.

Mr. Richmond said that the curve was always of the same shape, whatever its conditions, though its dimensions sometimes varied. The period during which no increase in acidity was indicated was determined by the point at which it was possible to detect acidity. Personally he did not think that less than ½ degree could be detected with certainty, and it was an appreciable time before so much acid as that was produced, so that the production of acid might be going on at its regular rate for some time before it was possible to know anything about it. They had had in mind the method of Dr. Lessing and Dr. Schryver, and intended to make use of it as soon as they could obtain the apparatus and had had sufficient experience of its use.

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THE ESTIMATION OF AMMONIA IN CARBONATED WATERS.

By G. D. ELSDON, B.Sc., A.I.C., and NORMAN EVERS, B.Sc., A.I.C.

(*Read at the Meeting, March* 6, 1912.)

During the examination of a series of samples of soda water, our attention was drawn to the smallness of the figures which we obtained for free ammonia, even in those waters which contained considerable quantities of albumenoid ammonia. Only four out of twenty samples contained any free ammonia at all, and of these 0·005 part per 100,000 was the highest. Some typical results are shown in Table I.

Table I.

Sample.	Parts per 100,000.	
	Free Ammonia.	Albumenoid Ammonia.
A	0·000	0·040
B	0·000	0·011
C	0·000	0·007
D	0·000	0·009
E	0·002	0·005
F	0·005	0·006

For the estimation of free ammonia, the contents of two syphons were transferred to a Winchester quart, the whole thoroughly shaken to remove the gas, and the ammonia estimated in the usual manner in 500 c.c. One c.c. of Nessler solution was used to 50 c.c. of distillate, the Nessler solution containing: Potassium iodide, 6·0 per cent.; mercuric chloride, 2·5 per cent.; potassium hydroxide, 15·0 per cent. (Frankland, "Thorpe's Dictionary of Applied Chemistry," vol. iii., p. 980).

In order to find out whether the presence of carbon dioxide in the distillate affects the estimation of free ammonia, a definite amount of standard ammonium chloride solution (1 c.c. = 0·00001 grm. ammonia) was added to an ordinary potable water known to be ammonia free. This water was then carbonated and the ammonia estimated as usual. Several similar estimations were carried out with results as set out in Table II.

Table II.

Sample.	Original Water.	After Carbonation.
A	0·004	0·001
B (1)	0·010	0·004
B (2)	0·010	0·005
C	0·020	0·003
D (1)	0·017	0·005
D (2)	0·017	0·007

The amount of ammonia found in each 50 c.c. of distillate was very unusual—*e.g.*, in the case of sample D (2) the first cylinder contained 0·000, the second 0·006, the third 0·001, and the fourth 0·000, suggesting that the carbon dioxide, which would be nearly all driven over into the first 50 c.c. of distillate, affects the colour produced by the Nessler reagent. We observed in some cases a transient yellow colour as the Nessler solution was added to the first 50 c.c. of distillate, but this entirely disappeared on shaking. On adding further quantities of Nessler solution to these cylinders, a permanent colour was produced in some cases; whilst in other cases, after a few moments' standing, a red precipitate appeared which proved to be mercuric iodide.

Effect of Carbon Dioxide on Nessler's Reagent.—Ten c.c. of Nessler solution were added to 500 c.c. of water containing 0·060 parts per 100,000 of free ammonia. The usual yellow colour was produced, but, on carbonating, this colour was immediately destroyed and, after a few minutes, precipitation of mercuric iodide began.

Two series of standards were made up, each containing 0·5, 1, 2, and 3 c.c. of standard ammonium chloride. The first was prepared with ordinary ammonia free water, and the second with carbonated ammonia-free water. It was found that on adding 1 c.c. of Nessler solution no colour was produced in any of the cylinders prepared with carbonated water, although the usual gradation was observed in the others; on adding a second c.c. of Nessler solution to the carbonated series, immediate precipitation occurred in all the cylinders.

From the above experiments it was quite obvious that the presence of carbon dioxide in quantity seriously affects the colour produced on adding Nessler solution to dilute solutions of ammonia. Our attention was then directed to the quantities of carbon dioxide which would thus interfere.

For this purpose a number of cylinders were taken containing a known fixed amount of ammonia and varying quantities of carbon dioxide, the latter being introduced by adding suitable amounts of standard acid and standard sodium carbonate. It was found that the presence of carbon dioxide in quantities exceeding about five parts by weight per 100,000 diminishes the colour produced, and that no colour is produced with quantities of carbon dioxide exceeding about 50 parts per 100,000. When the amount of carbon dioxide present approaches saturation, precipitation of mercuric iodide takes place in a short time. If a larger quantity of Nessler solution is used, the amount of carbon dioxide necessary to affect the colour is greater, but the tendency to precipitation is increased. It seems probable that the effect on the colour is caused by the reduction of the amount of caustic potash present, as hydrochloric acid in equivalent quantities has a somewhat similar action. The presence of sodium bicarbonate also interferes with this colour in precisely the same way as carbon dioxide. Sodium carbonate has no effect.

Modifications Attempted.—In order to get over the above difficulty, the effect of using a Nessler solution of greater alkalinity was tried. Although the results obtained by this method showed a distinct improvement, they were by no means satisfactory.

The removal of carbon dioxide by boiling with a slight excess of hydrochloric acid was then tried, the free hydrochloric acid being driven off by evaporation to dryness. The residue was dissolved in 500 c.c. of ammonia-free water, and the ammonia estimated as usual. The results were good, but the method was tedious.

Process Recommended.—Finally we adopted the following method: After removal of as much carbon dioxide as possible by shaking in a Winchester, 500 c.c. of the water are transferred to the distillation flask and 5 c.c. of $\frac{N}{1}$ sulphuric acid (or more if the alkalinity of the water requires it) are added. Fifty c.c. are then distilled off and rejected, thus removing the carbon dioxide. An equivalent quantity of standard sodium hydroxide is then added, and the usual amount of sodium carbonate. The estimation of free and albumenoid ammonia is then proceeded with in the usual manner. Using this process, we have examined a number of samples of water carbonated in the laboratory; the results are set out in Table III.

TABLE III.

Waters Carbonated in the Laboratory.

Source.	Ammonia Parts per 100,000.					
	Original Water.		Carbonated Water.			
			Ordinary Method.		Suggested Method.	
	Free.	Albumenoid.	Free.	Albumenoid.	Free.	Albumenoid.
Ammonia-free water	0·000	0·000	0·000	0·000	0·000	0·000
Upland surface water, raw	0·003	0·009	—	—	0·003	0·009
,, ,, ,, filtered ...	0·001	0·005	0·000	0·006	0·001	0·007
,, ,, ,, ,, ...	0·000	0·004	0·000	0·006	0·000	0·006
Stream water A	0·006	0·013	0·001	0·017	0·008	0·019
,, B	0·025	0·020	0·003	0·023	0·025	0·021
,, C, filtered	0·000	0·006	0·000	0·010	0·000	—
Deep well water	0·000	0·000	—	—	0·000	0·002
,, ,, filtered	0·000	0·004	0·000	0·008	0·000	0·008
Pond water A	0·065	0·065	0·055	0·065	0·065	0·065
,, B	0·055	0·045	0·017	0·045	0·055	0·045

From the above table it will be seen that the results obtained for free ammonia by the suggested process agree with those obtained on the uncarbonated water by the ordinary method. It is obvious that, if the first 50 c.c. of distillate obtained from a carbonated water by the ordinary method is divided or diluted for the purpose of Nesslerising, the amount of carbon dioxide present, and therefore the error, will be proportionately less.

The albumenoid ammonia is slightly increased by carbonation, whether it is estimated by the ordinary method or by the suggested method. This increase in albumenoid ammonia seems to take place immediately on carbonation, and no further increase takes place on standing. That no albumenoid ammonia is due to impurities in the carbon dioxide used is proved by the fact that, in the case of ammonia-free water (prepared by distillation with alkaline permanganate), no albumenoid ammonia was found after carbonation.

Acid Effluents.—It is sometimes necessary to estimate ammonia in effluents containing large amounts of free acid; this is usually done by making alkaline with sodium carbonate and distilling. It is evident that the carbon dioxide evolved may in some cases be sufficient to affect the estimation of ammonia, and to obtain a correct result the water must be made neutral with sodium hydroxide before adding sodium carbonate.

Commercial Soda-Water.—The following results were obtained with samples of commercial soda-water procured from ordinary sources:

TABLE IV.

Sample.	Ammonia Parts per 100,000.			
	Ordinary Method.		Suggested Method.	
	Free.	Albumenoid.	Free.	Albumenoid.
A	0·000	0·006	0·007	0·006
B	0·000	0·007	0·004	0·009
C	0·000	0·002	0·000	0·002
D	0·000	0·010	0·002	0·009
E	0·000	0·005	0·003	0·006
F	0·000	0·004	0·002	0·004
G	0·000	0·004	0·003	0·004
H	—	—	0·002	0·002

Note.—Since reading this paper we have studied the effect of carbon dioxide on Nessler solution prepared both according to the formula given in Fresenius, "Quantitative Analysis," vol. ii., p. 128, and according to that given in Sutton, "Volumetric Analysis," seventh edition, p. 465. Although the results obtained show an improvement on those given in our paper, owing to the larger amount of potash present, we find that carbon dioxide in amounts exceeding about fifty parts by weight per 100,000 causes a reduction in the colour obtained; this amount of carbon dioxide is much less than would be obtained in the first 50 c.c. of distillate from a carbonated water.

We have also compared the colour produced by the various Nessler solutions (made according to these formulæ) with a given quantity of ammonia, and find that there is very little difference between them.

DISCUSSION.

The **PRESIDENT** remarked that, as the Nessler solution contained a large excess of potash, which must already contain some carbonic acid, it seemed rather strange that a small extra quantity of carbonic acid should have so great an effect. If this action occurred in aerated waters which had been shaken to remove most of the carbonic acid, it must occur also in many natural waters which contained considerable quantities of carbonic acid capable of being given off on boiling, so that, apparently, such waters also should be acidified, and the carbonic acid boiled off, before the free ammonia was determined.

Mr. W. T. **BURGESS** said that he understood the authors to say that better results were obtained when the quantity of Nessler solution was increased. His own practice, particularly with waters likely to contain very little free ammonia, was to use, for the 100 c.c. Nesslerised, 6 c.c. of Nessler solution prepared according to the formula of Frankland, containing 62·5 grms. of potassium iodide per litre; and working in this way he had not noticed any diminution in the free ammonia due to carbonic acid. In carbonated waters as much as possible of the carbonic acid was of course got rid of before the ammonia determination was commenced.

Mr. E. M. **HAWKINS** suggested that it might be possible to avoid two distillations by adding an excess of recently ignited caustic lime, which would combine with the carbonic acid and render the addition of sulphuric acid unnecessary. From his own experience, he was inclined to think that the use of caustic lime would be attended by satisfactory results, and it would not, as far as he could see, cause any difficulty in the subsequent process of determining the albumenoid ammonia.

Mr. A. **CHASTON CHAPMAN** remarked that, while the authors had undoubtedly shown that some effect was produced on Nessler solution by carbonic acid, he was inclined to think that this effect would not have been noticeable had a more sensitive Nessler solution of the usual composition been used.

Mr. **EVERS**, in reply, said that the neutralisation of only about one-tenth of the potash present was sufficient to lessen the colour. He did not know whether the addition of caustic lime would be advantageous, but, as a matter of fact, the distillation was practically one continuous process. He should imagine that, if a Nessler solution containing more potassium iodide were used, the tendency towards precipitation would be greater. Dr. Thresh recommended a solution which gives, on adding it to the distillate, about the same strength as Sutton's as regards potassium iodide, but about one and a half times the strength of potash. With such a solution some colour, at any rate, would be obtained in most cases, but it would be reduced in many cases by the carbon dioxide, and in the ordinary way such a reduction might not be noticed.

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NOTE ON A NEW PRESERVATIVE FOR MILK, CREAM, ETC.

BY G. A. STOKES.

(*Read at the Meeting, March* 6, 1912.)

I HAVE recently examined a number of samples of milk which contained a new preservative, consisting of sodium nitrite, formaldehyde, and water, sold under the name of "Mystin" (*cf.* Monier-Williams, ANALYST, 1912, 155). This preparation, owing to the presence of sodium nitrite, fails to respond to Hehner's test and Schiff's reagent. In estimating the fat by the Gerber process, the presence of formaldehyde will generally be detected.

By adopting the following methods, as little as 2 drops of preservative per 100 c.c. of milk may be detected:

1. A portion of the milk is coagulated with dilute sulphuric acid, filtered, and a solution of diphenylamine in sulphuric acid added to the clear filtrate. The formation of a deep blue colour shows the presence of nitrite (or nitrate).

2. One hundred c.c. of the milk are acidified with 5 c.c. of strong phosphoric acid and distilled in a current of steam. About 10 c.c. are distilled into an equal volume of Schiff's reagent; if a pink coloration develops within five minutes the presence of formaldehyde is indicated. "Mystin" added to milk in the proportion of 3 c.c. per litre was found to be a fair preservative. Thus, after three days the acidity developed was only equivalent to 0·24 per cent. of lactic acid, whereas the same milk unpreserved showed an acidity equivalent to 0·69 per cent. of lactic acid.

DISCUSSION.

Dr. MONIER-WILLIAMS said that he was very glad to have heard Mr. Stokes's paper, because it directed attention to one or two new points. It was curious that the addition in this way of so little as 1 part of formaldehyde in 300,000 should be sufficient to preserve milk for three days. It was generally understood that, when formaldehyde was used as a milk preservative, a considerably larger quantity than this was required. It was interesting to note that potassium chlorate and potassium nitrate also interfered with the Hehner reaction. The action of these two substances might very likely depend on a direct oxidation of the colouring matter or of the formaldehyde, and possibly other oxidising agents might have a similar effect, if present in sufficient amount.

Mr. W. BACON remarked that possibly the increased antiseptic effect of the formaldehyde in this mixture might be due to the action of the nitrite in preventing condensation of the formaldehyde with the amino-acids of the milk proteids.

Mr. STOKES, in reply, said that he ought perhaps to have mentioned that his experiments were made during somewhat cold weather. Possibly in warmer weather the preservative effect might not have been so good. He had met with two cases in which this preservative was present in samples taken under the Sale of Food and Drugs Act.

THE ESTIMATION OF FERRIC IRON IN PRESENCE OF CERTAIN ORGANIC SUBSTANCES.

By JOHN THEODORE HEWITT, D.Sc., Ph.D., M.A., F.R.S., and GLADYS RUBY MANN.

(*Read at the Meeting, April* 3, 1912.)

Recently, one of the authors of this communication desired to measure the rate at which certain organic substances were oxidised by alkaline permanganate. It was an easy matter to stop the reaction at any instant by adding an excess of ferrous sulphate and dilute sulphuric acid; it was not, however, quite obvious how the amount of unchanged ferrous salt or of the ferric salt produced could be determined. Determination of unchanged ferrous salt is out of the question, as the residual organic matter may, and in many cases did, act as a vigorous reducing agent on acid solutions of dichromate or permanganate, hence a method for estimation of the ferric salt had to be adopted. Methods depending on the use of stannous or titanous chlorides had to be abandoned, as the organic substances in some cases contained nitro-groups, and consequently recourse was had to methods involving the use of thiosulphate.

The original method, due to Scherer, of considering the reaction finished when no further increase of the violet coloration was noticed on adding the thiosulphate to the ferric solution does not give very sharp results, so that the methods in which the reaction

$$2Fe^{\cdots} + 2S_2O_3'' = 2Fe^{\cdot\cdot} + S_4O_6''$$

is allowed to become complete, and the excess of thiosulphate is determined with iodine were examined. (Kremer and Landolt, *J. pr. Chem.*, 1861, **84**, 339; *Zeitsch. anal. Chem.*, 1862, **1**, 214; Oudemanns, *Zeitsch. anal. Chem.*, 1867, **6**, 129; 1870, **9**, 342; compare Balling, *ibid.*, **9**, 99.) The direct method of adding potassium iodide to the ferric solution,

$$2Fe^{\cdots} + 2I' = 2Fe^{\cdot\cdot} + I_2,$$

and titrating the liberated iodine with thiosulphate (F. Mohr, *Annalen*, 1860, **113**, 257; *Zeitsch. anal. Chem.*, 1863, **2**, 243; C. D. Braun, *J. pr. Chem.*, 1860, **81**, 423; *Zeitsch. anal. Chem.*, 1864, **3**, 452), would have been sufficiently accurate (see Joseph, *J. Soc. Chem. Ind.*, 1910, **29**, 187; Schatz, *Pharm. Zeit.*, 1910, **55**, 292) but for the fact that it was impossible to allow the iodine to remain in contact with the organic matter, which was frequently of unsaturated or phenolic character.

Experiments were consequently made with Oudemann's method, various organic substances being added at the same time. Generally 20 c.c. of an iron alum solution, about decinormal (5·585 grms. ferric iron per litre) in strength, were mixed with a solution of the organic compound under examination, then 3 c.c. of 1 per cent. copper sulphate and 1 c.c. of 1 per cent. ammonium thiocyanate added and $\frac{N}{10}$ thiosulphate solution run in until the colour of the ferric thiocyanate disappeared. Even with the copper salt as catalyst the end-point was never sharp, and the excess of thiosulphate was immediately titrated with standard iodine with starch as indicator.

Although the added organic substances are capable of reacting with iodine in many of the cases examined, the velocity of the reaction,

$$I_2 + 2S_2O_3'' = 2I' + S_4O_6'',$$

so far exceeds the speed at which the iodine is used by the organic substance that no difficulty was found in determining the end-point.

It must be admitted that better results are obtained when the operator has acquired practice; a very important point is not to use a greater excess of the thiosulphate than necessary, and to titrate this excess as rapidly as possible.

Mineral acid is added, if required, to break up feebly ionised salts (*e.g.*, ferric acetate); on the other hand, any large excess affects the estimation injuriously on account of the decomposition of the non-ionised thiosulphuric acid.

It is as well to standardise the thiosulphate on a ferric solution, since, if the standardisation be carried out with copper sulphate, the results obtained for iron appear to be rather low. The standard iron solution may be made by dissolving 48·22 grms. of iron-ammonia alum in water with the aid of 75 c.c. $\frac{N}{1}$ sulphuric acid and making up to 1 litre; if necessary, the iron may be estimated in this solution gravimetrically. The agreement which may be expected on titrating equal volumes (20 c.c.) of the same iron solution is shown in the following table (1 c.c. iodine = 1·003 c.c. thiosulphate):

	1.	2.	3.
Thiosulphate added	20·10	20·09	20·50
Iodine for back titration	0·35	0·40	0·76
Thiosulphate consumed by iron ...	19·75	19·69	19·74

The titration might conceivably be affected by—

1. The formation of a non-ionisable ferric compound or of a complex ferric anion.
2. Reduction of ferric to ferrous compound by the added organic substance.
3. Reaction of the iodine with the added organic substance as well as with the thiosulphate.

Whilst rapid work obviates any trouble due to the second or third causes, it intensifies that due to stability of non-ionisable ferric compounds. In the table of results it will be noticed that a very large excess of ammonium sulphate has the expected effect, which is not, however, great enough to make the titration useless unless very large amounts of the sulphate are present. Acetates give no trouble if mineral acid be added, acetic acid itself having but a slight influence. The deep colour produced by salicylates and salicylic acid interferes seriously, and the quantity of mineral acid which must be added to inhibit this is too great to allow of a subsequent safe titration with thiosulphate. The titration was practically unaffected by sugars and not greatly by tartaric acid or citric acid in presence of the amount of mineral acid (75 c.c. $\frac{N}{1}$ sulphuric acid per litre) used in making up the iron solutions.

In the table the results show the effect of the substances added. Twenty c.c. of the ferric solution (0·112 grm. iron) were first directly titrated, and the titration was then carried out with equal volumes of the same solution, the substance which might affect the result being added in gradually increasing quantities. The first column gives the name of the added substance, the second shows the weight thereof, and the third the volume in which the added substance was dissolved. In the fourth column the net volume of the thiosulphate employed in titrating the ferric iron is given—*i.e.*, the gross volume of thiosulphate added less the equivalent of the iodine used in the back titration.

Added Substance.			c.c. Thiosulphate used for Titration.
Name.	Weight. Grms.	Volume of Solution.	
Ammonium sulphate	0·000	0	19·73
	0·264	10	19·86
	0·528	20	19·59
	1·320	50	19·43
	2·640	100	18·72
Acetic acid	0·000	0	19·90
	0·240	10	19·88
	0·480	20	19·93
	1·200	50	19·52
	2·400	100	19·22
Sodium acetate ...	0·000	0	19·73
	0·545	10	13·55
	1·190	20	4·81
Tartaric acid ...	0·000	0	19·73
	0·150	10	19·56, 19·70
	0·300	20	19·65
	0·750	50	19·18
	1·500	100	18·74
Citric acid	0·000	0	19·73
	0·192	10	19·56
	0·384	20	19·54
	0·960	50	18·91
	1·920	100	15·85
Ethyl alcohol (96 per cent by volume).	0	—	19·90
	5 c.c.	—	19·93
	10 c.c.	—	19·73
	20 c.c.	—	19·68
Phenol	0·000	0	19·73
	0·094	10	19·91, 19·97, 19·94
	0·188	20	19·87
	0·470	50	19·82
	0·940	100	19·55
Mannitol	0·000	0	19·90
	0·182	10	19·91
	0·364	20	19·68
	0·910	50	19·54
	1·820	100	19·48

Added Substance.			c.c. Thiosulphate used for Titration.
Name.	Weight. Grms.	Volume of Solution.	
Glucose	0·000	0	19·73
	0·180	10	19·92, 19·84
	0·360	20	19·77
	0·900	50	19·52
	1·800	100	19·73
Galactose	0·000	0	19·90
	0·900	50	19·91
Cane-sugar	0·000	0	19·73
	0·338	10	19·87, 19·78
	0·676	20	19·75
	1·690	50	19·75
	3·380	100	19·72
Maltose	0·000	0	19·90
	1·800	50	19·52
Lactose	0·000	0	19·90
	1·800	50	19·81
Dextrin (British gum).	0·000	0	19·73
	0·162	10	19·87, 19·89
	0·324	20	19·78
	0·810	50	19·79
	1·620	100	19·51

In conclusion, we desire to record our thanks to Mr. J. L. Baker for his kindness in supplying us with very pure specimens of maltose, lactose, and galactose.

East London College.

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ON THE RELATION BETWEEN THE KIRSCHNER AND POLENSKE VALUES FOR MARGARINES CONTAINING COCOANUT OR PALM KERNEL OILS.

BY E. RICHARDS BOLTON, H. DROOP RICHMOND,
AND CECIL REVIS.

(*Read at the Meeting, April* 3, 1912.)

IN a paper published last year (ANALYST, 1911, **36**, 333), it was pointed out that the determination of the Kirschner value would enable the presence of small quantities of butter-fat to be estimated in the presence of cocoanut oil, and, further, that in itself it furnished a reliable figure for the percentage of cocoanut oil in the absence of butter-fat. The figures then given for the Kirschner values for mixtures containing no butter-fat fell practically on a straight line, but when the mixture contained butter-fat up to 10 per cent. a somewhat irregular curve was obtained. We have reinvestigated this point, and find that the irregularity was due to the fact that the values obtained for mixtures containing up to 15 per cent. of cocoanut oil were carried out with an Irish butter of very low Reichert-Meissl value (22), while the other values were obtained by using a butter of much higher Reichert-Meissl value (30); and we had not at that time recognised how sensitive to the presence of butter the Kirschner value was. We have therefore redetermined the values for those mixtures which contained the butter of low Reichert-Meissl value, with a butter of more normal figure, and are now able to make the following deductions:

1. That for the Kirschner values for both cocoanut and palm kernel oils (with or without admixture of butter-fat up to 10 per cent.) a straight line can be plotted which will represent, with very great closeness, the values experimentally obtained (see curves).

Further, for any percentage of cocoanut or palm kernel oils the difference in the Kirschner values for "no butter-fat" and for any percentage of butter-fat (up to 10 per cent.) will be proportional within very small limits to the percentage of butter-fat; the closeness of the agreement indicates that the relation will also hold for higher percentages of butter-fat.

2. On examining the values obtained both for cocoanut and palm kernel oils, it was seen that the Polenske value was practically independent of the amount of butter-fat present, when present up to 10 per cent. in the mixture, and is practically dependent on the presence of the cocoanut or palm kernel oil only. The mean value was therefore calculated from the four values obtained for mixtures corresponding to each different percentage of cocoanut or palm kernel oil (see Table I.), and on plotting these mean values a regular curve was obtained. The following equations have been worked out for these "mean value" curves:

$$x\,(\text{C.N.O.}) = 12{\cdot}3\,(\text{P} - 0{\cdot}45)^{0{\cdot}747};$$

$$x\,(\text{P.K.O.}) = 16{\cdot}72\,(\text{P} - 0{\cdot}45)^{0{\cdot}806};$$

where $x =$ the percentage of cocoanut or palm kernel oil.

TABLE I.

Cocoanut Oil.		Butter-Fat.			
		0 per Cent.	2 per Cent.	5 per Cent.	10 per Cent.
0 per cent.	Reichert-Meissl ...	0·38	0·92	1·70	3·25
	Polenske	0·31	0·38	0·42	0·55
	Kirschner	0·18	0·81	1·49	2·70
5 per cent.	Reichert-Meissl ...	0·87	1·66	2·16	4·10
	Polenske	0·71	0·79	0·68	0·93
	Kirschner	0·22	0·74	1·31	2·85
10 per cent.	Reichert-Meissl ...	1·64	2·39	3·40	4·86
	Polenske	1·17	1·20	1·37	1·26
	Kirschner	0·32	0·88	1·58	2·99
15 per cent.	Reichert-Meissl ...	2·67	3·16	4·27	5·52
	Polenske	1·73	1·80	1·69	2·01
	Kirschner	0·38	0·94	1·69	2·99
25 per cent.	Reichert-Meissl ...	3·91	4·85	5·53	6·54
	Polenske	2·91	2·61	2·96	2·93
	Kirschner	0·54	1·15	1·94	3·08
50 per cent.	Reichert-Meissl ...	6·05	6·60	7·70	9·10
	Polenske	7·06	7·16	6·90	7·30
	Kirschner	1·10	1·56	2·27	3·37
75 per cent.	Reichert-Meissl ...	6·97	8·34	9·20	10·50
	Polenske	12·36	11·91	12·20	12·30
	Kirschner	1·50	1·91	2·57	3·68
100 per cent.	Reichert-Meissl ...	8·08	—	—	—
	Polenske	16·02	—	—	—
	Kirschner	2·01	—	—	—

These relations show that the influence on the Polenske figure of 1 per cent. of cocoanut oil is the same as that of about 1·75 per cent. of palm kernel oil. The figures obtained from the equations for palm kernel oil are almost identical with the experimental figures, but the agreement is not quite so good in the case of cocoanut oil, and we have not taken into account the value for 100 per cent. in calculating the equation to this curve. The values in the latter case show somewhat irregular fluctuations, which are probably due to the distillation of the volatile insoluble acids in cocoanut oil, on account of their lower molecular weight than those of palm kernel oil, being more influenced by the other constituents of the mixture. The chief difference is in the value for cocoanut oil itself, the experimental value being 16·5 as against 17·05 calculated from the formula. We are, after many experiments, of the opinion that it is not possible to obtain the true value for cocoanut oil in the ordinary apparatus, and this point will be referred to later.

We therefore point out that the Polenske value acts as an "indicator," so that, when a margarine containing cocoanut or palm kernel oils is examined by the Reichert-Meissl-Polenske-Kirschner process, reference to this mean curve at once determines the percentage of cocoanut or palm kernel oil apart from other values. The corresponding Kirschner value obtained from the typical curve then determines the presence or absence of butter-fat, the Reichert-Meissl value acting as a confirmatory figure, and controlling the small fluctuations which may occur in the Kirschner value occasioned by a variation in butyric acid content of different butters.

The tables (II. and III.) here given for cocoanut and palm kernel oils, with and without admixture of butter-fat, are the typical values obtained from the curves which are also given, and we feel that they will prove a reliable guide provided that the method be carried out under standard conditions laid down in the paper previously referred to, the standard apparatus being also employed (ANALYST, 1904, 29, 155).

On account of the similarity of the results obtained for cocoanut and palm kernel oil, the following formula will give the percentage of butter present with either fat for the Kirschner and Polenske values found experimentally:

$$\text{Butter per cent.} = \frac{K - (0{\cdot}262\,P^{0{\cdot}63} + 0{\cdot}09)}{0{\cdot}242};^{*}$$

or nearly as exactly by the more simple formula:

$$\text{Butter per cent.}\ \frac{K - (0{\cdot}1\,P + 0{\cdot}24)}{0{\cdot}244.}$$

The following formulæ connect the Kirschner value and percentage of butter-fat when neither cocoanut nor palm kernel oil is present:

$K = 0{\cdot}236\,B + 0{\cdot}33$, or, with a small increase in the probable error, $K = 0{\cdot}244\,B + 0{\cdot}28$, which is practically the formula given above.

Exception may be taken to the above formulæ, on the ground that it supposes a certain fixed value for the butyric acid content of the butter present. While this is undoubtedly true, the variations so caused will not in the vast majority of cases lead to errors of any great magnitude, and the percentage of butter determined by the formula has usually been within 0·5 per cent. of that actually present.

We think it necessary to draw attention to an observation we have made recently. In the standard apparatus, that portion of the still-head which passes through the cork and into the interior of the distillation flask is provided with a small hole in the side to prevent the collection of condensed liquid in the still-head.

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[*] Values of $(0{\cdot}262\,P^{0{\cdot}63} + 0{\cdot}09)$:

P.	Value.	P.	Value.
0·5	0·26	8	1·06
1	0·35	9	1·13
2	0·50	10	1·21
3	0·61	11	1·28
4	0·72	12	1·34
5	0·81	13	1·41
6	0·90	14	1·47
7	0·98	15	[illegible]

As originally designed by Polenske, this hole had a fixed distance from the stopper of the flask. Insufficient attention is paid to this point by makers of the apparatus, and we have found that if the hole is much more than 1 cm. from the lower surface of the cork, low Polenske values may be obtained with high percentages of cocoanut oil; and we think that if this method be made a standard one, particular attention should be given to this point.

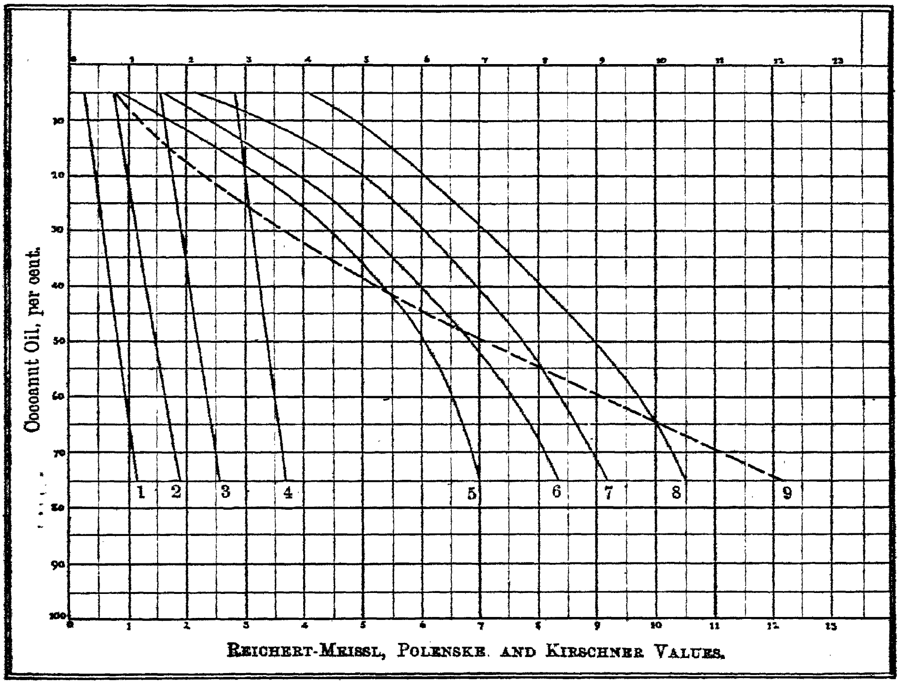
TABLE II.

Cocoanut Oil.	Polenske Indicator Value.		Butter-Fat.			
			0 per Cent.	2 per Cent.	5 per Cent.	10 per Cent.
0 per cent.	0·45	Kirschner ...	0·18	0·80	1·49	2·70
		Reichert-Meissl ...	0·38	0·92	1·70	3·25
5 per cent.	0·76	Kirschner ...	0·25	0·75	1·55	2·82
		Reichert-Meissl ...	0·87	1·65	2·15	4·10
10 per cent.	1·22	Kirschner ...	0·34	0·84	1·60	2·90
		Reichert-Meissl ...	1·60	2·45	3·42	4·90
15 per cent.	1·75	Kirschner ...	0·42	0·92	1·68	2·96
		Reichert-Meissl ...	2·52	3·15	4·35	5·55
25 per cent.	2·91	Kirschner ...	0·60	1·08	1·82	3·08
		Reichert-Meissl ...	3·92	4·57	5·55	6·55
50 per cent.	7·10	Kirschner ...	1·02	1·50	2·20	3·38
		Reichert-Meissl ...	6·05	6·88	7·72	8·95
75 per cent.	12·19	Kirschner ...	1·45	1·92	2·55	3·70
		Reichert-Meissl ...	7·00	8·35	9·20	10·50
100 per cent.	16·5	Kirschner ...	1·88	—	—	—
		Reichert-Meissl ...	8·08	—	—	—

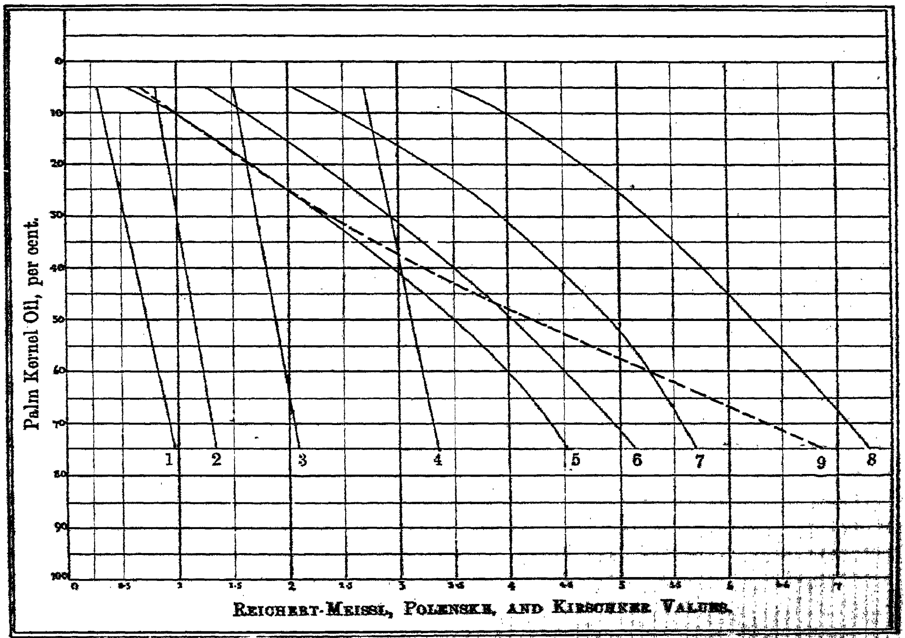
THE RELATION BETWEEN THE KIRSCHNER AND POLENSKE VALUES FOR BUTTER-FAT.

As the Kirschner value for butter is almost entirely obtained from the butyric acid present, it is evident that a relation between the Polenske value and the Kirschner value will be more sensitive than the relation, at present accepted, between the Reichert-Meissl and Polenske values. We tentatively put forward the following table with the hope that other workers will criticise or verify the figures:

Kirschner Value.		Polenske Value.
20	... ...	1·60
22	... ...	2·10
24	... ...	2·65
26	... ...	3·20



1. Kirschner, 0 per cent. butter.
2. ,, 2 ,, ,,
3. ,, 5 ,, ,,
4. Kirschner, 10 per cent. butter.
5. R.-M., 0 per cent. butter.
6. ,, 2 ,, ,,
7. R.-M., 5 per cent. butter.
8. ,, 10 ,, ,,
9. Mean Polenske.



1. Kirschner, 0 per cent. butter.
2. ,, 2 ,, ,,
3. ,, 5 ,, ,,
4. Kirschner, 10 per cent. butter.
5. R.-M., 0 per cent. butter.
6. ,, 2 ,, ,,
7. R.-M., 5 per cent. butter.
8. R.-M., 10 per cent.
9. Mean Polenske.

A variation of ± 1 must be allowed in the Polenske value corresponding to any particular Kirschner value, but it would appear that less than 5 per cent. of cocoanut oil will cause the Polenske value to fall outside this limit, except in those cases in which cows have been specially fed for the purpose of upsetting the Polenske process. In conclusion we desire to express our thanks to Messrs. G. A. Payne and H. R. Burnett for carrying out the experimental part of the investigation.

TABLE III.

Palm Kernel Oil.	Polenske Indicator Value.		Butter-Fat.			
			0 per Cent.	2 per Cent.	5 per Cent.	10 per Cent.
0 per cent.	0·45	Kirschner ... Reichert-Meissl ...	0·18 0·38	0·80 0·92	1·49 1·70	2·70 3·25
5 per cent.	0·68	Kirschner ... Reichert-Meissl ...	0·30 0·53	0·80 1·27	1·51 2·05	2·70 3·50
10 per cent.	1·00	Kirschner ... Reichert-Meissl ...	0·35 1·00	0·85 1·62	1·54 2·50	2·75 4·00
15 per cent.	1·35	Kirschner ... Reichert-Meissl ...	0·40 1·35	0·90 2·00	1·57 2·90	2·80 4·35
25 per cent.	1·97	Kirschner ... Reichert-Meissl ...	0·48 1·97	0·97 2·60	1·65 3·62	2·87 4·97
50 per cent.	4·22	Kirschner ... Reichert-Meissl ...	0·72 3·50	1·16 4·05	1·87 4·92	3·12 6·22
75 per cent.	6·87	Kirschner ... Reichert-Meissl ...	0·97 4·55	1·35 5·15	2·07 5·70	3·35 7·30
100 per cent.	9·82	Kirschner ... Reichert-Meissl ...	1·07 5·22	— —	— —	— —

DISCUSSION.

Mr. W. BACON asked whether, in the form of apparatus with a very large distilling tube, this tube was insulated by some non-conducting material? If not, since the tube was so close to the neck of the flask, a great deal of condensation would be likely to occur.

Mr. E. M. HAWKINS asked whether, in a mixture consisting of cocoanut oil and of butter, of which the Kirschner figure was known, it would be possible, from the Polenske and Kirschner figures, to determine with a fair degree of certainty the proportion of cocoanut oil?

Mr. REVIS said that with the special tube described there was no condensation at all, the distillation being, as a matter of fact, more rapid than with the ordinary form. He should like once again to emphasise the importance of always using the same standard pattern of apparatus. The exact details did not matter, provided they were the same in every case. If that were so, the results obtained by different workers would agree, and the results obtained by each would be consistent and concordant among themselves. They (the authors) had found that the Polenske, Reichert-Meissl and Kirschner processes were all three quite as accurate and definite as an ordinary titration of acid and alkali. The question asked by Mr. Hawkins as to cocoanut oil and butter mixtures involved some difficulty, because butter varied so greatly as regards its proportion of butyric acid. As they had pointed out, a much more sensitive relationship was shown between cocoanut oil and butter fat when the Kirschner and Polenske figures were used than when the Reichert-Meissl and Polenske figures were used. They put forward their results quite tentatively, but believed the process to be capable of showing the presence of as little as 3 per cent. of cocoanut oil in butter fat. There must always be some doubt as to the exact quantity of butter fat present in a mixture, because of the varying nature of the butter itself.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Chemical Valuation of Digitalis. J. Burmann. (*Bull. Soc. Chim.*, 1912, **11**, 221.)—The author has already shown (*Bull. Soc. Chim.*, 1910, **7**, 973) that in Keller's method of judging of the value of digitalis by the examination of its active glucoside, digitoxin, it is only a pseudo-digitoxin that is estimated, and his results have been confirmed by Kraft (*Schweiz. Woch. Chem. Pharm.*, 1911, 12-13). For the estimation of the total glucosides soluble in chloroform, 100 grms. of the liquid, obtained by dialysing the digitalis with 20 per cent. alcohol, are mixed with 60 grms. of absolute alcohol, and made up to 190 grms., with 50 per cent. alcohol. A mixture of 30 grms. of basic lead acetate solution (sp. gr. 1·240) and 30 grms. of absolute alcohol is then added, the liquid stirred and filtered, and 125 grms. of the filtrate (=50 grms. of the dialysate) treated with hydrogen sulphide, and filtered from the lead sulphide through a Buchner's funnel. The precipitate is washed two or three times with 50 per cent. alcohol, and the filtrate and washings concentrated under reduced pressure (preferably below 50° C.) to about 50 c.c. to expel the alcohol, after which 2 c.c. of 10 per cent. ammonia solution are added, and the liquid extracted with chloroform for one and a half to two hours. The chloroform extract is filtered into a tared 200 c.c. Erlenmeyer flask, the solvent evaporated on the water-bath, and the residue dried at 100° C. in a current of dry air. It is next taken up with 3 grms. of chloroform, the solution mixed with 7 grms. of absolute ether, and the glucosides reprecipitated by the addition of 50 grms. of petroleum spirit. After standing for some hours, the supernatant liquid is decanted and replaced by the same quantity of petroleum spirit, the flask being well shaken and allowed to stand. Finally, the liquid is again decanted, and the residue dried on the water-bath in a current of dry air until constant in weight. The glucosides thus separated are in the form of an amorphous white powder, and give the reactions typical of Keller's digitoxin. They may be crystallised by dissolving 0·03 to 0·04 grm. in 1 c.c. of absolute alcohol and adding 0·5 c.c. of water. After standing for several hours the deposit will show under the microscope acicular crystals grouped in rosettes (ψ-digitoxin or Kraft's hydrodigitalin) and prismatic tables (digitoxin). The crystals may be separated by means of their different solubilities in ether and water, and identified by their melting-points (145° to 150° C., and 247·5° C. respectively). In this way the following results were obtained in the examination of four dialysates of digitalis:

	1. Per Cent.	2. Per Cent.	3. Per Cent.	4. Per Cent.
Total glucosides	0·152	0·148	0·118	0·111
Glucosides by Keller's method ...	0·118	0·116	0·091	0·085
Digitoxin	0·034	0·032	0·027	0·026
Proportion of digitoxin	22·5	21·6	22·0	23·4

Physiological experiments upon animals showed that the glucosides soluble in chloroform were more toxic than Keller's digitoxin.

C. A. M.

Baking Qualities of Flour. Willard and Swanson. (*Chem. News*, 1912, **105**, 97-99.)—Failure to correlate the behaviour of flours with their gliadin-gluten ratios suggested that the differences observable might be caused by substances present in very small amounts. The production of a good loaf depends on the physical properties of the gluten, and these may be much affected by small quantities of associated substances. Such substances may also operate in quite a distinct manner—viz., by favouring or inhibiting the growth of the yeast, and among these would be the amino acids produced from the wheat proteins by the hydrolytic action of the yeast. The addition of unextracted bran to flour had a notably deteriorating effect on the loaf as regards both volume and texture. If the bran were previously extracted with cold or hot water the effect was less marked on loaf texture, and the addition of bran extract to the flour was very favourable. The fact that extracted bran and bran extract each produced better results than when entire bran is used was unexpected but confirmed on repetition. Figures are given showing the effect of various substances on loaf volume. Extracts derived from wheat scourings were, on the whole, undesirable, causing sticky doughs; peptones had no effect till present in large quantity, and were then deleterious as they produced marked stickiness. Glycocoll was markedly bad in every way, the dough being sticky, runny, and stringy, and resembling that from badly germinated wheat; the loaf volume was reduced and the texture impaired. Leucin produced results like glycocoll, but less pronounced; it caused the development of a disagreeable odour. Aspartic acid proved slightly favourable, but asparagin was injurious. The latter, while shorten-ing the time of rising (probably by stimulating the growth of the yeast), weakened the gluten and so decreased the oven expansion and loaf volume. Similar effects are observed with flour from germinated wheats, and asparagin is known to be among the products of accompanying protein metabolism (*cf.* Ford and Guthrie, ANALYST, 1908, **33**, 323). Ammonium chloride had a most beneficial effect even in small traces, and appeared to assist yeast growth, as the time of rising is shortened. Ammonium acetate had a similar but less marked effect, but in larger amounts impaired the texture. Ammonium tartrate and phosphate had but little effect. Sodium bicarbonate was detrimental alike to time of rising and volume of loaf, while sodium phosphate, sodium formate, and potassium nitrate call for no special comment. A detailed study of the effects of germination of wheat on the resulting flour was made, material being prepared from corns germinated for from 1 to 5 days. Up to 3 days little effect was produced, but after this time bad effects at once became apparent; texture of loaf, volume, crumb, and alcohol-soluble protein all being adversely affected. The deleterious influence of flour from germinated wheat was strongly manifested even when only 1 part in 30 of sound flour was employed, while with larger amounts the product showed that the mixtures were quite unfit for bread-making. H. F. E. H.

Detection of Fluorine in Foods, etc. A. Sartori. (*Chem. Zeit.*, 1912, **36**, 229-230.)—For the estimation of fluorides in wine or fruit-juice, according to the author, 200 c.c. of the sample are treated with a few drops of 20 per cent. sodium sulphate solution and 10 c.c. of 10 per cent. barium acetate, and the mixture is allowed to stand for twelve hours. The precipitate is collected on a filter, incinerated and tested for fluorides by the waxed watch-glass method. The limit of sensitiveness by this method is about 0·3 mgrm. of fluorine. J. F. B.

Comparative Study of Methods for Determination of Hard and Total Soft Resins in the Hop. H. V. Tartar and C. E. Bradley. (*J. Ind. Eng. Chem.*, 1912, **4**, 209-213.)—It is shown that Siller's method (ANALYST, 1909, **34**, 485), though useful enough with Continental hops, leads to over-estimation of the soft resins when applied to seeded hops such as are grown in America or England. This is due to the extraction of fatty oil from the seeds (*cf.* Briant and Harman, *J. Inst. Brew.*, 1910, **16**, 5), which are crushed when the sample is put through a mincing machine as directed by Siller. On the other hand, Briant and Meacham's method (*J. Fed. Inst. Brew.*, 1897, **3**, 233) gives very much lower results for soft resins, and correspondingly higher ones for hard resins, than does the latest form of Lintner's volumetric method (ANALYST, 1909, **34**, 53; 1910, **35**, 352), or a new method described in the present paper. Lintner's original method (*Zeitsch. f. d. ges. Brauw.*, 1898, **21**, 407) gives results which accord well with those obtained by the method of Briant and Meacham, but both methods are believed to be faulty in that they fail to extract the whole of the soft resin. A hop which shows only 8 per cent. of soft resin when subjected to Briant and Meacham's process or the original Lintner process may yield 16 per cent. when analysed in accordance with Lintner's more recent proposals or by the authors' method. Lintner's new method differs from the old one only in the fine grinding of the sample, to which no objection can be taken when the final determination is to be made by volumetric process dependent on the acid function of the soft resins.

The new method described is as follows: A 10 grm. sample of hops is extracted with ether for eight to ten hours. The ethereal extract thus obtained is filtered, and the filtrate made up to 200 c.c. Half this volume is used for the determination of total resins, the other half for the determination of soft resins. For the determination of total resins, 100 c.c. is placed in a 250 c.c. flask and evaporated at about 40° C. to very small bulk, the last of the ether being removed in a vacuum desiccator at ordinary temperature. The residue is taken up in alcohol, filtered free from wax, and the filtrate made up to 100 c.c. Of this 100 c.c., a portion of 20 c.c. (equivalent to 1 grm. of hops) is transferred to a tared beaker, and the alcohol removed by evaporation in a vacuum oven at 50° C. For the determination of soft resins the remaining 100 c.c. of the ether extract is taken down to dryness exactly as in the determination of total resins. The residue is taken up with about 100 c.c. of petroleum ether (b.p. 40° to 45° C.), stirring with a rod and leaving to stand some time. The hard resin is then removed by filtration, the petroleum ether removed from the filtrate exactly as the ether was removed at an earlier stage, and the residue taken up with alcohol. The alcoholic solution is filtered from wax, made up to 100 c.c., and 20 c.c. (equivalent to 1 grm. of hops) is transferred to a tared beaker, and evaporated and weighed as in the determination of total resins. When dried in the manner described, there is much less change of composition—as judged by the absence of browning—than when resort is had to higher temperatures, and a constant weight is obtained, which is never the case when a temperature of 80° C. is employed.

The authors believe their method to be the most accurate available, but they consider Lintner's modified method to be the best method for commercial purposes, as it is rapid, and yields results which seldom differ by more than a few tenths of 1 per cent. from those yielded by their own method. Even with 10 grm. samples the sampling error cannot with certainty be kept below 0·3 per cent. G. C. J.

Tests for Linseed Oil and its Adulterants. J. F. Liverseege and G. D. Elsdon. (*J. Soc. Chem. Ind.*, 1912, **31**, 207-208.)—The following modification of the well-known Livache test for quantitatively expressing the drying-power of oils has been found to yield uniform results: Litharge, finely-powdered and passed through a No. 40 sieve, is spread out in a thin layer in an incubator at 20° to 22° C. overnight to become constant in weight. About 10 grms. of this litharge are introduced into a flat-bottomed German silver dish, 3 inches diameter and 1 inch deep, 0·7 to 0·9 grm. of oil added, and after weighing, 5 c.c. of methylated ether (sp. gr. 0·720) are run into the mixture, and the litharge spread out in a uniform layer by thoroughly rocking the dish. The dish and contents are kept in the incubator at 21° C. and weighed after one, two, or more days, until the weight is either constant or diminishing, and the weight gained then expressed in terms of percentage of the oil taken. The added ether is volatilised after half an hour, and leaves an inappreciable residue. By this test, raw linseed oil ceases to gain weight in two days, and then usually loses weight. With boiled oils, the oxidation is usually complete in one day. The following are typical figures:

	One Sample.	Per Cent. Maximum.	Minimum.
Raw Linseed Oils.			
Gain after two days	17·4	18	15
,, ,, three days	16·9	—	—
,, ,, fourteen days ...	15·9	—	—
Boiled Linseed Oils.			
Gain after one day	14·1	14	12
Gain after two days	13·6	—	—

Soya Oil.—Gain = 8·4 to 8·9 per cent.

Cotton-seed Oil.—Gain = 6·6 per cent.

Rape Oil and Arachis Oil.—Gain = 2·5 per cent.; after twenty-one days, rape oil, 5·7; arachis oil, 4·1 per cent.

Olive Oil (2 samples).—Gain = 0·6 and 1·4 per cent. in two days; after twenty-one days, 2·5 and 3·4 per cent. respectively.

Resin Oil.—Loss = 0·3 per cent. in one day; then gain = 9·9 per cent. in two days, and 19·4 per cent. in thirteen days.

Thus, drying oils do not gain after about two days, while non-drying oils may continue to gain for weeks. The gain in weight of oils of a particular class varies with the iodine value, and there is evidence of some relationship between these figures for oils of different classes. A. R. T.

Chemical Composition of some New Zealand Meat Extracts. A. M. Wright. (*J. Soc. Chem. Ind.*, 1912, **31**, 176.)—Moisture, organic matter, mineral salts, chlorine, fat, and total nitrogen were determined by the methods described by the author (*J. Soc. Chem. Ind.*, 1907, **26**, 1229); acidity by titration with $\frac{N}{10}$ potassium hydroxide and phenolphthaleïn and calculating as lactic acid; insoluble and coagulable proteins (*U.S. Dept. of Agriculture, Bureau of Chem.*, *Bull.* 107, p. 115); proteoses by the zinc sulphate method (*ibid.*); peptone-like bodies and polypeptides by precipitation, with the proteoses, by the tannin salt-reagent, their amount being found by difference (*J. Amer. Chem. Soc.*, 1906, **28**, 1485); nitrogen as total meat bases by deducting the nitrogen present as ammonia from the nitrogen in the tannin salt filtrate; ammonia by the magnesium oxide method (*U.S. Dept. of Agriculture, Bureau of Chem.*, *Bull.* 107, p. 9); purin bases by Schittenhelm's method (*ibid.*, *Bull.* 90, p. 129); creatin and creatinin by Folin's colorimetric method (*Zeitsch. physiol. Chem.*, 1904, **41**, 223); phosphoric acid and potash by examination of the ash (*U.S. Dept. of Agriculture, Bureau of Chem.*, *Bull.* 107, pp. 3, 4, and 11).

A "burned" flavour and a dark colour impair the commercial value of an extract; they are shown, by the analyses given of six samples, to be accompanied by undue amounts of peptone-like substances, intermediate between the peptones and the amino acids. The characteristic salts of meat extract are potassium dihydrogen phosphate and potassium monohydrogen phosphate; in general, the mineral constituents show great variations. O. E. M.

Acid-Soluble Phosphorus Compounds of some Feeding Materials. Hart and Tottingham. (*Twenty-Seventh Annual Report of the Agric. Exp. Sta. Univer. Wisconsin*, 95-106; through *Inter. Inst. Agric.*, 1912, 3, 398.)—Phytin, which is a complex organic phosphorus salt of potassium, magnesium, and calcium, is widely distributed, particularly in seeds, as a reserve material. It is present in the entire seeds of maize, oats, and barley, but is largely localised in the outer layers of the wheat seed; it could not be isolated from swedes or alfalfa (Lucerne) hay. Phytin phosphorus forms from 40 to 50 per cent. of the total phosphorus in the aforementioned seeds. In alfalfa which was cut at the most favourable stage of gro for forage, the inorganic phosphorus formed 63 per cent. of the total; there is also present about 17 per cent. of phosphorus in some unknown organic combination.

H. F. E. H.

Estimation of Saccharin in Foods. G. Possetto and G. Issoglio. (*Giorn. Farm. Chim.*, 1912, **61**, 5-11; through *Chem. Zentralbl.*, 1912, I., 754.)—The separation of the saccharin from the chief constituents of foods by dialysis is recommended. In the case of milk, jams, etc., a portion of the sample is rendered alkaline with sodium carbonate and submitted to dialysis for twenty-four hours; at the end of this time the dialysed solution is acidified with phosphoric acid, and the saccharin shaken out with ether. The dialysed solution obtained in a similar way from cocoa should be decolorised with animal charcoal before being shaken out with ether.

W. P. S.

Composition of Scammony Root and of Scammony Resin. F. B. Power and H. Rogerson. (*J. Chem. Soc.*, 1912, **101**, 398-412.)—Scammony root contains a small quantity of sucrose, an essential oil and furfuraldehyde, crystalline scopoletin and its glucoside, 3-4-dihydroxy-cinnamic acid, fatty acids, and a resin containing ipuranol, $C_{23}H_{38}O_2(OH)_2$, melting at 285° to 290° C.; a-methyl-butyric and tiglic acids; jalapinolic acid and its methyl-ester; valeric acid; and traces of other substances. Scammony gum-resin contains tiglic acid, a valeric acid, jalapinolic acid and its methyl-ester, and glucosides, dextrose, etc. The resins obtained from the root and the commercial gum-resin are thus similar in character, but not identical. A comparison of the resin from scammony-root with that obtained from the root of *Ipomœa orizabensis* (*J. Chem. Soc.*, 1912, **101**, 1-17), shows that the two products differ considerably in composition. Both the resins are complex in character, but consist to a large extent of the glucosides and methyl-pentosides of jalapinolic acid, $C_{15}H_{30}(OH)(CO_2H)$ and its methyl-ester. While, however, the methyl-pentose obtained from the hydrolysis of the resin of scammony root appears to be identical with rhamnose, that from the resin of *I. orizabensis* yields a new crystalline tetra-acetyl derivative. The resin from the last source contains also small amounts of hentriacontane, $C_{31}H_{64}$, and cetyl alcohol, which are not present in the resin from scammony root, and marked differences are observed in extracting the crude resins and the products of their alkaline hydrolysis with various solvents. The names by which these complex resins have hitherto been known, such as "jalapin" or "scammonin" should now be discarded, as they are not individual substances.

A. R. T.

Inversion of Sucrose by the Enzymes of Honey. O. Achert. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 136-139.)—Results of experiments carried out by the author show that honey which has not been heated above 55° C. is capable of inverting added sucrose; the inversion proceeds at the ordinary temperature and is more or less rapid. For instance, a mixture of honey and sucrose was prepared containing 22·0 per cent. of the latter; after the lapse of four months the mixture was found to contain only 2·2 per cent. of sucrose. When the honey has been heated to a temperature of 100° C., it loses its power of inverting sucrose. The quantity of free acids in the honey does not play any part in the process, as neutralised honey inverts sucrose at the same rate as does natural honey. W. P. S.

Estimation of Tannin in Wines. P. Malvezin. (*Bull. Soc. Chim.*, 1912, **11**, 300-303.)—The method described previously (ANALYST, 1911, **36**, 412) may be modified slightly in order to render the estimation more rapid, the chief alteration being that the zinc-tannin precipitate is collected on a filter immediately after the wine and precipitant have been boiled for the prescribed period of five minutes. After the precipitate has been washed, it is dissolved in dilute sulphuric acid and the solution titrated with $\frac{N}{10}$ permanganate as described. The factor 0·120 should be employed for calculating the quantity of gallotannic acid from the volume of permanganate solution required for the titration, and the titration should be continued until a pink coloration is obtained which does not disappear within three minutes.

W. P. S.

Detection of Small Quantities of Zinc in Wine. A. Straub. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 140.)—The following process is described for the detection of small amounts of zinc in wines, for instance, in wines which have been treated with potassium ferrocyanide and a zinc salt in the fining operation (*cf.* ANALYST, 1905, **30**, 264). From 100 to 200 c.c. of the wine are boiled, hot sodium carbonate solution is added, and the mixture heated for some length of time; the precipitate is then collected on a filter, washed, dissolved in a small quantity of hydrochloric acid, and the solution oxidised by heating with potassium chlorate. An excess of sodium acetate is now added, the solution is boiled, filtered to remove iron and aluminium hydroxides, etc., and the filtrate then tested with hydrogen sulphide.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Microscopic Differentiation of Hemp and Flax Fibres. P. Sonntag. (*Ber. Deutsch. botan. Ges.*, 1912, **29**, 669; through *Chem. Zentralbl.*, 1912, I., 1058.)—With the microscope focussed on the upper surface, flax shows a right-hand spiral forming an average angle of 10·21 degrees with the cell axis. With a deeper focus, a left-hand spiral of a steeper twist is observed. Hemp shows the same double spiral, but at an average angle of only 3·7 degrees. In both cases the structure is only observed in thin bast cells after boiling in water or acetic acid. Observation is best carried out on the broken ends of a torn fibre. O. E. M.

Seventh Report on Research Work of the Metropolitan Water Board. A. C. Houston. 1912.—This Report consists of the following six sections: 1. Search for pathogenic organisms in raw river water. 2. The comparative vitality of "cultivated" and "uncultivated" typhoid bacilli in artificially infected samples of raw river water. 3. The comparative vitality of the typhoid bacillus in raw Thames water at different temperatures. 4. On the biological characters of *B. coli* isolated from (1) raw river water, (2) stored water, and (3) stored and filtered water. 5. On the advantages of passing raw river water through small reservoirs antecedent to storage in large reservoirs. 6. On the advantages of occasionally using precipitation methods antecedent to the storage of raw river water in large reservoirs. Among the conclusions arrived at by the author are the following: 1. The typhoid bacillus and Gärtner's bacillus are not habitually present in small amounts, such as 10 c.c., of raw river water; and since the water will ultimately be stored for several weeks and then filtered, the margin of security is very great, in view of the extremely high death-rate of uncultivated typhoid bacilli when added artificially to river water. 2. In the Sixth Research Report (ANALYST, 1911, **36**, 185) it was shown in a "girl carrier" case that "uncultivated" typhoid bacilli perish much more rapidly than "cultivated" organisms. A "male carrier" case amply confirms the previous finding, and it may be taken as true that thirty days' storage of river water is tantamount to sterilisation so far as water-borne epidemic disease germs are concerned. 3. Laboratory experiments lead to the conclusion that the typhoid bacillus lives longer at low (32° to 41° F.) than at higher (50° to 98·6° F.) temperatures. For seven months in the year the mean river temperature of about 58° F. is specially unfavourable to the sustained vitality of the typhoid bacillus; during the remaining five months of the year the mean temperature of about 41° F. is much less favourable to the rapid death of this organism. Even at freezing-point the reduction of typhoid bacilli by the second week was over 99 per cent., and by the fourth week only one survived out of 3,029 added initially. In the case of the Metropolitan water-supply the author is of opinion that even in the coldest months of the year four weeks' storage of river water prior to filtration affords reasonable, if not absolute, protection from water-borne diseases. 4. This report is of a highly technical character, and deals with the physiological differentiation of numerous varieties of *B. coli*, using the direct plating-out method not preceded by cultivation in an enrichment liquid medium. 5 and 6. The author confirms his previous opinion (Third Research Report) as to the desirability of passing the *raw* river water through small reservoirs before storage. On three separate occasions, when samples were examined before and after such treatment, the percentage reduction, when averaged, was as follows: Bacteria, 36, ammoniacal nitrogen 15, albumenoid nitrogen 5·2, permanganate 7, turbidity 19, colour 8. The pre-storage treatment may be advantageously supplemented by the use of coagulants such as sulphate of alumina or alumino-ferric, and among other benefits derivable is the possibility of utilising the worst quality of flood water, and so preventing the depletion of the storage reservoirs under any conditions save those of drought. H. F. E. H.

Sterilisation of Water and Dairy Materials by Ultra-Violet Rays. M. Ringelmann. (*J. d'Agric. Prat.*, 1911, **75**, 619-621; through *Inter. Inst. Agric.*, 1912, **3**, 266.)—Ultra-violet rays from a mercury lamp traverse quartz or rock crystal, but not glass and thin layers of air. Water must be free from matter in suspension; the sterilisation on an industrial scale of liquids like wine, beer, milk, or broth, which contain colloid substances, is not to be expected, owing to the low penetrative qualities of the rays in such media. The walls of the lamp should not be in direct contact with water to be sterilised, since the yield of rays falls off in proportion as the temperature of the lamp is lowered. On a laboratory scale milk has been successfully sterilised by allowing films not exceeding a few millimetres in thickness to flow between quartz plates, the rate of flow being regulated by the inclination of the plates. Water for butter-making is favourably affected, an impure sample after treatment causing a prolongation in the life of butter made with it from eight days to a month. The most useful application of the rays is in the sterilisation of drinking water. H. F. E. H.

ORGANIC ANALYSIS.

Detection of Tertiary Alcohols. M. Guerbet. (*Comptes rend.*, 1912, **154**, 713-715.)—A means of distinguishing between primary, secondary, and tertiary alcohols is based upon the differences in their behaviour when heated with potassium hydroxide (*cf. Comptes rend.*, 1911, **153**, 1487). A mixture of 3 c.c. of the alcohol and 3 grms. of potassium hydroxide (previously dehydrated by fusion) is heated for sixteen hours at 230° C. in a sealed tube. After cooling, the point of the tube is broken under water, and the gases collected and measured. In the case of primary or secondary alcohols the amount of gas will be considerable, whereas with tertiary alcohols it will only amount to a few c.c. at most, or there may be absorption of water into the sealed tube. The solid product of the reaction is then treated with water. If the alcohol was primary or secondary, it will have been completely transformed into the potassium salt of the corresponding acid, and will be entirely soluble, whereas in the case of secondary or tertiary alcohols an oily layer of about the same volume as the original alcohol will be formed upon the surface of the water. C. A. M.

Aromatic Grass Oils. Part II. (*Bull. Imp. Inst.*, 1911, **9**, 333-340.)—Part I. of this article (ANALYST, 1912, 61) dealt with citronella oils, and the present part is devoted to lemon-grass oils. These are derived mainly from the two grasses, *Cymbopogon flexuosus* and *C. citratus*, the former yielding oils soluble in two or more volumes of alcohol (70 per cent.), and the latter giving, as a rule, oils not completely soluble in even ten volumes alcohol (90 per cent.). An oil from *C. pendulus* (India) also yields an "insoluble" oil.

Ceylon Lemon-grass Oils.—Three samples distilled from *C. citratus* gave the following results:

	1.	2.	3.
Yield of oil, per cent.	0·22	0·25	0·37
Sp. gr., 15°/15° C.	0·892	0·891	0·893
Opt. rotation, 100 mm. at 20° C.	−0·42°	−0·65°	+1·12°
Citral by sodium bisulphite, per cent.	75·0	74·0	76·0
Solubility	Not completely soluble even in 10 vols. of 70, 80, or 90 per cent. alcohol.		

Two other oils examined, stated to be *flexuosus* oils, were found to be mixtures of such oil with the oil from *C. nardus* (citronella grass), and gave abnormal results.

Indian Lemon-grass Oils.—Four samples yielded results as follow:

	"Tyrna" Oil.	"Cochin" Oil.	"Mariani" Oil.	"Ceylon" Oil.
Odour	—	Fine	—	—
Sp. gr., 15°/15° C.	0·902	0·905	0·914	0·906
Opt. rotation, 100 mm. at 20° C.	-0·7°	-0·65°	-0·1°	-0·1°
Citral, by sodium bisulphite, per cent.	72·5	84·5	73·0	76·0
Solubility in 70 per cent. alcohol	Not completely sol. in 10 vols.	Sol. 1: 2. More alcohol produced slight turbidity.	Not completely sol. in 10 vols.	
Solubility in 90 per cent. alcohol	Sol. in quantities up to 1 vol.; cloudy on addition of more alcohol.	Sol. in all proportions; excess of alcohol produced slight turbidity.	Sol. in quantities up to 1 vol.; turbid on addition of more alcohol.	

The "Cochin" lemon-grass oil thus belongs to the group of "soluble" oils, and is exceptionally rich in citral. Most commercial lemon-grass oils contain from 70 to 75 per cent. of citral.

Uganda Lemon-grass Oils.—The following are typical of six oils (from *C. citratus*) examined from this source:

	1 (Entebbe).	2 (Entebbe).	3.
Sp. gr., 15°/15° C.	0·872	0·881	0·894
Opt. rotation at 20° C. ...	-0·12°	+0·17°	-0·4°
Aldehydes by sodium bisulphite, per cent.	64·5	70·5	75·0
Solubility in alcohol	Not completely sol. in 10 vols. of 80 per cent.	Not completely sol. in 10 vols. of 80 per cent.	Insol. in 10 vols. of 70 per cent.

Bermuda Lemon-grass Oil.—A sample of this oil from *C. citratus* showed on analysis: Sp. gr., 15° C., 0·8689; optical rotation at 20° C., -0·35°; citral, 40 per cent.; not completely soluble in 70 per cent., or in 80 per cent. alcohol. The percentage of citral is very low.

Montserrat Lemon-grass oil is of the insoluble type, and one sample examined contained over 74 per cent. of citral.

A. R. T.

Detection of Carotin. M. Tswett. (*Ber. Deut. Botan. Ges.*, 1912, **29**, 630-636. Through *Chem. Zentralbl.*, 1912, I., 951.)—For colouring matters belonging to the same group as carotin the name *carotinoids* is suggested. For the detection of carotin an alcoholic or petroleum spirit extract of the colouring matter is first obtained, and its elective solubility in a two-phase system of 80 per cent. alcohol and petroleum spirit is estimated. Should a portion of the colouring matter be electively soluble in the petroleum spirit it is possibly carotin. After removal of all alcohol the absorption capacity of the petroleum spirit solution is then tested with calcium carbonate, and should the solution retain its colour in the presence of an excess of the absorbent agent, its spectrum should be examined. The positions of the two principal absorption bands in the spectrum of carotin are somewhat differently placed by different observers. In the author's experiments with carotin derived directly from the leaves these bands occurred at 492 to 475 $\mu\mu$ and 460 to 445 $\mu\mu$ respectively. With regard to the micro-chemical detection of carotin, it was found that the potassium hydroxide method of Molisch (*Ber. Deut. botan. Ges.*, 1896, **14**, 18) and the author's resorcinol method (*Botan. Centralbl.*, **81**, 83) did not give entirely definite reactions. In the latter method the tissue containing chlorophyll is treated with concentrated resorcinol solution, which dissolves the plasmatic proteins and lipoids. If, then, about 1 per cent. of hydrogen potassium phosphate be added, the lipoids agglomerate into green globules and there separate, here and there, bundles of yellow crystals, which also contain red crystals. These masses of crystals consist of xanthophyllene and carotin. C. A. M.

Errors in the Estimation of Cholesterol by the Ritter Method; Effect of Autolysis on Cholesterol. H. J. Corper. (*J. Biol. Chem.*, 1912, **11**, 37; through *Chem. Zentralbl.*, 1912, I., 1055.)—Ritter's method of estimating cholesterol (*Zeitsch. f. physiol. Chem.*, 1901, **34**, 430) was investigated, pure cholesterol and ox-spleen being used; it was found that the use of sodium ethoxide causes unavoidable errors. With too little, the esterification of the fats is incomplete, and the amount of cholesterol is over-estimated; with too much, the cholesterol cannot be completely extracted from the salt-mixture by ether, and too little is found. The cholesterol content in the spleen of the dog is not materially altered by autolysis. Ox spleen contains about 0·4 per cent. of cholesterol calculated on the moist substance. O. E. M.

Estimation of Citric Acid. D. S. Pratt. (*U. S. Department of Agriculture, Bureau Chem.*, through *Chem. Eng.*, 1912, **15**, 72-74.)—The following method yields satisfactory results in the examination of fruit juices, etc., containing citric and other organic acids in admixture. The citric acid is oxidised to acetone dicarboxylic acid, and the acetone produced is weighed as a mercury sulphate addition-product. Fifty grms. of the juice are treated with 110 c.c. of alcohol (95 per cent.) to remove pectin bodies. The liquid is filtered after fifteen minutes and the residue washed with more of the alcohol. The solution is diluted to an alcoholic strength of about 50 per cent., and barium acetate solution (20 per cent. in water) added to precipitate all the citric acid. After stirring, allowing the precipitate to settle, filtering the solution and thoroughly washing the precipitate on the paper with alcohol (50 per cent.) to remove sugar, the paper and its contents are dried until alcohol is removed. (Ether may be used in place of alcohol at this stage for washing the precipitate.) About 50 c.c. of water and 3 to 5 c.c. of syrupy phosphoric acid are added to the barium citrate, which, on warming, completely dissolves, when the liquid is filtered and the paper and residue washed till the solution measures 100 c.c. An aliquot part of this solution, containing about 0·05 to 0·15 grm. citric acid, is measured into a distilling flask, 5 to 10 c.c. of phosphoric acid added together with 400 c.c. of hot water. When briskly boiling, potassium permanganate solution (0·5 grm. per litre) is run in from a dropping funnel at the rate of 1 to 2 drops per second till the pink coloration is permanent throughout the liquid. The apparatus used by the author consists of a distillation-flask (500 c.c. capacity) attached to a spiral condenser, the former carrying a funnel, the stem of which extends ½ inch through the stopper, and which is drawn out to a small bore. The distillation-flask contains a glass rod, to the lower end of which is fused a short piece (½ inch) of small tubing, the device helping to produce steady ebullition. As the permanganate is added drop by drop, the acetone produced by oxidation of the citric acid is distilled off as soon as formed, and further oxidation prevented. The receiver contains 30 to 40 c.c. of Denigès' reagent, prepared by adding, with constant stirring, 200 c.c. of strong sulphuric acid to 50 grms. of mercuric oxide and 500 c.c. of water, heating, if necessary, on the water-bath till solution is complete, and filtering the liquid after cooling and diluting to 1 litre. The distillation is continued till only 50 to 100 c.c. of solution remain undistilled, when the solution in the receiver is boiled gently under a reflux-condenser for three-quarters of an hour after the liquid becomes milky. The solution is filtered hot through a Gooch crucible, washed with water, alcohol, and finally ether, and dried in the water-oven for half an hour. The weight of the precipitate, which is, according to Denigès, $(2HgSO_4.3HgO)_3.4CH_3.CO.CH_3$, multiplied by 0·22, gives the amount of citric acid in the fruit juice taken. In many cases precipitation of barium citrate and its subsequent solution in phosphoric acid are unnecessary, but sugar-containing products should be examined as described, since sugar retards the oxidation of the citric acid. Malic and tartaric acid do not interfere with the process. Duplicate determinations agree within 2 or 3 mgrms. of citric acid with the quantities specified. The factor given (0·22) does not correspond with any simple formula, and has been arrived at as a mean of a number of experiments. A. R. T.

Analysis of Textile Finishes. E. Schmidt. (*Chem. Zeit.*, 1912, **36**, 313-315.) —The author proposes a scheme for the identification of finishing materials in textiles. The finish is dissolved in as concentrated a form as possible by extraction with organic or inorganic solvents. Fats, resins, and mineral substances are estimated in separate samples. The substance extracted from the cloth and free from fat is dissolved in water. The aqueous solution is treated with a few drops of nitric acid, and the separation of a gelatinous precipitate would indicate the presence of norgin. The precipitate is warmed slightly and filtered; the filtrate is cooled and tested with a neutral solution of ammonium molybdate. If a white flocculent precipitate is formed, either at once or after a short time, the presence of glue is indicated.

After standing, the solution is filtered, neutralised with ammonia, and treated with an excess of alcohol. The formation of a precipitate may indicate the presence of starch, dextrin, gums, plant mucilages, or norgin; dextrose and glycerol remain in the filtrate. J. F. B.

Quantitative Estimation of Hydroxy-, Amino-, and Imino-Derivatives of Organic Compounds by Means of the Grignard Reagent, and the Nature of the Changes taking Place in Solution. H. Hibbert. (*J. Chem. Soc.*, 1912, **101**, 328.)—Hibbert and Sudborough's method (*J. Chem. Soc.*, 1904, **85**, 933) gives low results with most hydroxy-compounds if phenetole is the solvent used; the fatty alcohols up to and including propyl alcohol behave quite abnormally in both phenetole and amyl ether. Aromatic and most fatty hydroxy-compounds give accurate results in amyl ether, alone or in admixture with ketones, esters, etc. In phenetole, but not in amyl ether, aromatic amines give low results. Hibbert and Sudborough's method (*J. Chem. Soc.*, 1909, **95**, 477) for the estimation of amino- and imino-groups is not applicable to the lower fatty amines, as these form stable additive compounds with the magnesium methyl iodide, with the result that gas is not evolved, or only slowly, and on heating. Pyridine appears to be the most suitable solvent for hydroxyl compounds, and primary and secondary amines, but investigation of the lower fatty alcohols and fatty amines in this respect has not been carried out. Dimethylaniline may apparently also be employed. O. E. M.

New Method for the Separation of Tertiary from Secondary and Primary Amines. H. Hibbert and A. Wise. (*J. Chem. Soc.*, 1912, **101**, 341.)—The primary and secondary amines form additive compounds with magnesium alkyl halides which immediately decompose at ordinary temperatures, with evolution of the corresponding fatty hydrocarbon:

$$(1)\ R{\cdot}NH_2 + MgR'I = R{\cdot}NH(MgI) + R'H$$

$$(2)\ R_2 : NH + MgR'I = R_2 : N\ (MgI) + R'H$$

while tertiary amines, having no available hydrogen atom, yield additive compounds incapable of this decomposition. To the mixed amines, alone or dissolved in ether, is added excess of an ethereal solution of magnesium methyl iodide or bromide, the ether distilled off, and the product heated to 200° to 280° C. in an oil-bath, when the tertiary amine distils over. The other amines are recovered by steam distillation from sodium hydroxide. O. E. M.

Elm-Seed Fat. A. Pawlenko. (*Chem. Rev. Fett- u. Harz-Ind.*, 1912, **19**, 43-44.)—The seeds of the elm-tree contain about 45 per cent. of kernels, which on extraction with petroleum ether yield a greenish-yellow fat (26·1 per cent.) belonging to the cocoanut-oil group. A sample thus extracted gave the following values: Sp. gr. at 20° C., 0·9559; solidification-point, 3·5° C.; melting-point, 5·7° C.; acid value, 5·57; saponification value, 277·3; Hehner value, 75·45; Reichert value, 3·75; and iodine value, 32·2. The fat contained 56·2 per cent. of capric acid with smaller quantities of butyric and oleic acids, and 14·82 per cent. of glycerol. The shells of the nuts also yielded about 2 per cent. of a hard yellow wax-like fat, melting at 59·5° C. and having a saponification value of 187. The kernel fat had a pleasant taste and odour and was apparently well suited for the purposes of food. It also yielded a hard soap which was particularly soluble in cold salt water, but which separated on the surface on heating the salt solution to 78° C. The extracted residue contained 7·9 per cent. of nitrogen, and ought, therefore, to be a valuable feeding stuff. C. A. M.

Characteristics of Cotton Wax. C. Piest. (*Zeitsch. angew. Chem.*, 1912, **25**, 396-399.)—A sample of crude American cotton (ash, 2·44 per cent.; Schwalbe's copper value, 3·57) yielded the following amounts of extract to different solvents: Ether, 0·74; benzene, 0·87; petroleum spirit, 0·50; and alcohol, 1·20 and 1·27 per cent. The alcoholic extract had a saponification value of 159, and iodine value of 22·1. In the case of nine samples of normal cotton purified for nitration the following range of values was obtained: Copper value, 0·65 to 1·47; ash, 0·12 to 0·27; wood-gum, 0·32 to 0·90; ether extract, 0·09 to 0·37; carbon tetrachloride extract, 0·12 to 0·32; and alcoholic (absolute) extract, 0·24 to 0·53 per cent. The cotton wax in the normal purified wool was soluble to the extent of about 50 per cent. in absolute alcohol, whilst about 25 per cent. was soluble in ether and in carbon tetrachloride. In respect of its melting-point, saponification value, and iodine value, it closely resembled Japan wax. When present in cotton wax it has the effect of increasing the copper value. C. A. M.

Refractometric Estimation of the Strength of Formaldehyde Solutions. L. T. Reicher and F. C. M. Jansen. (*Chem. Weekblad.*, 1912, **9**, 104-109; through *Chem. Zentralbl.*, 1912, I., 949.)—Experiments with Zeiss's immersion refractometer have shown that the refractometric reading is a linear function of the concentration of a formaldehyde solution. If 15·5 be subtracted from the reading, and the difference be divided by the factor 3·17, the result will give the percentage of formaldehyde in the solution. C. A. M.

Hübener's Bromide Method for the Estimation of Caoutchouc. F. W. Hinrichsen and E. Kindscher. (*Chem. Zeit.*, 1912, **36**, 217-218, 230-232.)—Hübener's method for the estimation of caoutchouc in vulcanised rubber (ANALYST, 1909, **34**, 170 and 365) by bromination in presence of water has been criticised by various authors, and recently in an adverse manner by Esch (ANALYST, 1911, **36**, 558). The present authors had already shown that the estimation of the combined bromine after oxidation with nitric acid involves serious losses of bromine, which, however, may be avoided if the substance be oxidised by fusion with alkali carbonates. It is now shown, however, that if this source of error be eliminated the method devised by Hübener becomes fundamentally unsound, and invariably yields results which are much too high. The investigations of the authors on mixtures of known composition lead to the following conclusions: (1) In the case of soft vulcanised rubbers, complete bromination is very difficult to attain, because the sample cannot be ground sufficiently fine; there is no means whereby the final point of the bromination in presence of water may be recognised. (2) The precipitated bromide always contains an excess of bromine above the theoretical quantity, owing to the occlusion either of bromine or brominated compounds. The method of purifying the bromide by washing with alcohol and carbon tetrachloride, recommended by Hübener, is quite insufficient. (3) Superbromination invariably takes place, and the caoutchouc values calculated from the combined bromine are too high. This error is only partially compensated by the opposing error due to loss of bromine when the compound is oxidised by boiling with nitric acid in presence of silver nitrate. (4) The estimation of the uncombined sulphur in the aqueous filtrate from the bromide precipitate is unreliable; considerable quantities of the combined sulphur are continuously oxidised and are estimated in the aqueous filtrate. (5) Consequently, the percentage of vulcanisation sulphur found in the bromide is always too low. (6) The theoretical basis of Hübener's method, differentiating unvulcanised caoutchouc, caoutchouc monosulphide and disulphide, is therefore left at present without experimental support. J. F. B.

Source of Error in the Kjeldahl-Gunning Method. P. A. W. Self. (*Pharm. J.*, 1912, **88**, 384-385.)—The author finds that considerable loss of ammonia may take place by volatilisation from the boiling acid liquid when the temperature rises above a certain point, and that this loss is markedly accelerated as the liquid approximates to the composition $KHSO_4$. In some experiments made, practically the whole of the ammonia was lost from highly concentrated potassium sulphate and sulphuric acid mixtures. It is pointed out that there is a limit to the amount of organic substance which may be used in an experiment in order to avoid loss of nitrogen, and approximately this is, where 25 c.c. of sulphuric acid and 12 grms. of potassium sulphate are employed, not more than 3·5 grms. of a carbohydrate material, 3·0 grms. of a fatty product containing from 5 to 10 per cent. of oil or fat, and 1·5 grms. containing 80 to 90 per cent. of oil or fat. At least 15 grms. of sulphuric acid should remain at the end of the heating period. A. R .T.

Estimation of the Molecular Weight of Small Quantities of Fatty Acids. W. Arnold. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 129-135.)—Full details are given for estimating the molecular weight of small quantities of water-soluble and other fatty acids by titration with potassium hydroxide solution and determining the weight of the resulting soap. An essential part of the process consists in powdering the soap residue before it is dried, it having been found that the last traces of water cannot be expelled from a compact mass of soap. A quantity of the fatty acid is placed in a flask and neutralised by the addition of alcoholic $\frac{N}{10}$ potassium hydroxide solution; in the case of solid fatty acids, a portion of the potassium hydroxide is added, and the contents of the flask warmed to dissolve the fatty acid, the final part of the titration being carried out after cooling the solution, as slightly more alkali is required for the neutralisation when the solution is hot. Phenolphthaleïn is used as the indicator, and the amount of solid matter introduced by this solution must be estimated separately and an allowance made. The soap solution is then transferred to a weighed platinum basin, evaporated slowly, and the residue dried for forty-five minutes in a water-oven. The residue is now broken up into a fine powder by the aid of a small spatula, and the drying continued for a further two and a half hours. The molecular weight is then calculated from the formula:

$$M = \frac{(s - v \times f) \times 20000}{v}$$

where M is the molecular weight, s the weight of the soap, v the number of c.c. of $\frac{N}{20}$ potassium hydroxide solution used, and f the potassium equivalent $\left(\frac{39-1}{20000}\right)$ of each c.c. of the latter solution; should the potassium hydroxide contain traces of salts, their quantity must be estimated by sulphating a definite volume of the solution, and correcting the equivalent accordingly. Results of experiments with a series of fatty acids, ranging from butyric acid to stearic acid, show that the method yields correct figures; in over 90 per cent. of the analyses the figures found agreed within 2 units of those required by theory. W. P. S.

Method for Determining the Relative Reactivity of Organic Compounds. H. Hibbert. (*J. Chem. Soc.*, 1912, **101**, 341.)—The total theoretical quantity of methane is not in general evolved on the interaction of a hydroxy-compound and an excess of magnesium methyl halide in phenetole; the amount is not affected by the presence of ketones, esters, etc. If, however, there is insufficient Grignard reagent, it will be apportioned between the hydroxy-compound and the ketone, according to their relative reactivity for it. To 0·2880 grm. (1 mol.) of α-naphthol was added 3 mols. of the ketone, 6·0 c.c. of phenetole, and 5·0 c.c. of magnesium methyl iodide in phenetole, this being the quantity necessary to react with all the α-naphthol. The methane evolved was measured, and the reactivity-factor calculated as the theoretical volume evolved by 0·2880 grm. of α-naphthol, divided by that found. The known greater reactivity of fatty over aromatic ketones is reflected in the table of results on a series of ketones. O. E. M.

Estimation of Oxalic Acid in the Leaves of Conifers. J. Otto. (*Zeitsch. anal. Chem.*, 1912, **51**, 296-300.)—Having observed by microscopic examination that the leaves of conifers contained apparently varying quantities of calcium oxalate crystals, the amount varying with the age of the leaves, the author estimated the quantity actually present. Pine leaves (needles) one year old were found to contain from 0·2 to 0·9 per cent. of oxalic acid, whilst older leaves contained up to 2·3 per cent. W. P. S.

Preliminary Notes on the Examination of Various Stick-Lacs and Shellacs. J. H. Hoseason and O. Klug. (*J. Soc. Chem. Ind.*, 1912, **31**, 165.)—Five kilogrms. of the stick-lac were ground, sifted through a 40-mesh sieve, and mixed; 5 grms. were macerated for twenty-four hours in warm distilled water, filtered, and washed. The dried filtrate is the coloured water extract. The dried residue was macerated with 90 per cent. alcohol, the extract evaporated to a syrup and dropped on fat-free cotton-wool, and rapidly extracted, after the alcohol had been driven off, with cold ether. The ether was removed by evaporation with alcohol, the liquid made up to 50 c.c. with alcohol, and 5 c.c. taken for (ether-soluble) resin and two portions of 20 c.c. for two Hübl tests. The residue in the cotton-wool was dissolved out with alcohol to 100 c.c., and 5 c.c. taken for (ether-insoluble) resin, and 40 c.c. for two Hübl tests. To insure drying, the film of resin substance in determining the resin must be very thin. The residue from the treatment with water and alcohol was extracted with chloroform; the wax obtained on evaporating off the chloroform contained a brown substance, included in the wax content. The final residue (woody fibre, insect remains, and probably protein colour compounds) was dried and weighed. Analyses of a series of stick-lacs, button-lacs, and shellacs are given. The necessity for a standard method of carrying out the Hübl test is emphasised.

O. E. M.

New Method for the Determination of the Reducing Sugars. E. C. Kendall. (*J. Amer. Chem. Soc.*, 1912, **34**, 317-341.)—Briefly, the method consists in modifying Fehling's reagent by substituting potassium carbonate for caustic soda, and salicylic acid for Rochelle salt. The cuprous oxide is filtered off, dissolved in hot nitric acid, and the copper determined by the modified iodide method as previously described by the author (ANALYST, 1912, 66). Potassium carbonate is shown to be the most satisfactory alkali, and gives more copper than sodium carbonate with the same weight of sugar. Rochelle salt causes some spontaneous reduction of copper, and the citrate solution proposed by Benedict (*J. Biol. Chem.*, 1908, **5**, 485) is found to be inaccurate owing to depression and variation caused in the reducing power of the sugar in its presence. An alkaline salicylate solution replacing the alkaline tartrate was found to give no spontaneous reduction of copper even after seven hours heating in the boiling-water bath, and changes in the weight present had but very small effect on the reducing power of the sugars. It is not possible to replace salicylic acid by sodium salicylate, although the acid must be present in the alkaline solution as the potassium salt. Numerous experiments are described in which variations in procedure were made in order to obtain optimum conditions. For the preparation of standard tables by the new method, specially purified sugars were used, and their purity carefully checked by the polariscope and otherwise. The author prepared maltose by the barley diastase method of Baker (ANALYST, 1908, **33**, 393); the other sugars were purchased and recrystallised. The method of reduction was as follows: The sugar employed was measured out in solution of appropriate strength into a 200 c.c. Erlenmeyer flask, and the total volume made up to 100 c.c. Five grms. of dry salicylic acid were then added, followed by 15 c.c. of copper sulphate solution (equivalent to 2 grms. of $CuSO_4 5H_2O$) and 25 c.c. potassium carbonate solution (equivalent to 15 grms. of the carbonate) without any agitation of the flask. This order must be adhered to. The flasks are now shaken with a rotary motion. As soon as the salicylic acid has dissolved, the flask is immersed in the boiling-water bath for twenty minutes. The cuprous oxide is filtered on a perforated porcelain dish covered with asbestos felt, and finally dealt with by the author's method alluded to above. The sugars for which tables are given are glucose, invert sugar, lactose, and maltose.

H. F. E. H.

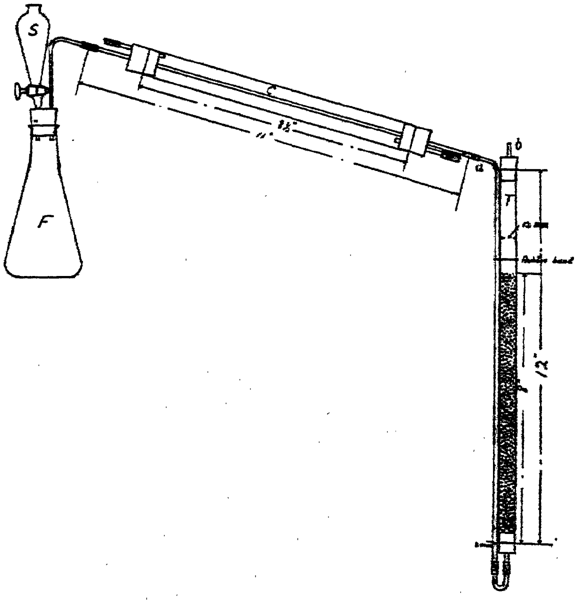
INORGANIC ANALYSIS.

Separation and Estimation of Arsenic and Antimony. G. Bressanin. (*Ann. chim. anal.*, 1912, **17**, 81-84.)—Blattner and Brasseur have proposed to estimate quantities of arsenic such as occur in commercial mineral acids as follows: Hydrochloric acid, if of lower sp. gr. than 1·16, is first brought up to this density by addition of pure sulphuric acid (sp. gr. 1·45), and 50 c.c. are taken for the test. Iodide of potassium (5 c.c. of a 30 per cent. solution) is added, and the arsenious iodide filtered off on asbestos and washed with a few c.c. of pure hydrochloric acid containing 10 per cent. of potassium iodide. The precipitate is then dissolved in water, the solution saturated with sodium bicarbonate, and the sodium arsenite titrated with $\frac{N}{10}$ iodine. Sulphuric acid, if free from tin and lead, is diluted until its sp. gr. is 1·45, and treated similarly. In presence of lead or tin it is first diluted with an equal bulk of pure hydrochloric acid. The author finds that the method underestimates arsenic in hydrochloric acid by 2 per cent. of the amount present, but that it is quite satisfactory with sulphuric acid free from lead or tin. In presence of these metals hydrochloric acid must be added, but its amount must not exceed one-third of that of the sulphuric acid, or the results will be low. The washing with hydrochloric acid containing potassium iodide is said to do more harm than good, and it is found better to reduce the amount of potassium iodide employed for precipitation to 2 or 3 c.c. The arsenic in hydrochloric acid may be estimated exactly by mixing the acid with twice its volume of sulphuric acid (sp. gr. 1·45), adding 2·5 c.c. potassium iodide solution, filtering, washing with a mixture of pure hydrochloric acid (sp. gr. 1·16) and pure sulphuric acid (sp. gr. 1·45) in the proportions 1 : 2, and proceeding as already described. The iodide reaction affords a very sensitive qualitative test for arsenic in mineral acids, a faint but distinct yellow opalescence appearing instantly if even so little as one part in a million be present.

The author has extended the method to the estimation of antimony in sulphuric acid. The optimum concentration of acid is that which corresponds to a sp. gr. of 1·5 or a little higher. Potassium iodide is added, and the precipitate washed with pure acid of the same density, and then redissolved by means of a 10 per cent. solution of Rochelle salt. Since antimonious iodide is soluble in concentrated hydrochloric acid, the method is not applicable to that acid, but the same circumstance affords a basis for the separate estimation of arsenic and antimony. The substance is dissolved in sulphuric acid (sp. gr. 1·5), and the solution made up to a definite volume with acid of this strength. In one aliquot portion arsenic and antimony are estimated together in the manner described for antimony, whilst, in another, arsenic is estimated alone after adding to the sulphuric acid solution one-third of its volume of hydrochloric acid, which prevents the precipitation of antimonious oxide. G. C. J.

Short Method for Determination of Soluble Arsenic in Commercial Lead Arsenates. B. E. Curry and T. O. Smith. (*J. Ind. Eng. Chem.*, 1912, **4**, 198-201.)—The method of the American Association of Official Agricultural Chemists involves the shaking of 2 grms. of the sample with 2 litres of water eight times daily for ten days. It is shown that identical results may be obtained by continuous stirring for eighteen hours. The authors find it more convenient to work with 500 c.c. of water, but the results thus obtained do not accord with those obtained by the official method, since lead arsenate itself is soluble to some extent in water. If 0·45 be added to the percentage of arsenic acid found when using only 500 c.c. of water for extraction, the result thus corrected will agree exactly with that obtained by the official method. But since 2 litres of water at 20° C. dissolve as much lead acetate as corresponds to 0·6 per cent. of arsenic acid in a 2 grm. sample, and since commercial lead arsenate contains only from 0·1 to 3 per cent. (usually about 0·3 per cent.) of free arsenic acid, it is suggested that the official method should require the extraction to be made at a fixed temperature, say 20° C., and that from the apparent percentage of arsenic acid found there should be made a deduction at the rate of 0·03 for each 100 c.c. of water used for extraction. G. C. J.

Volumetric Determination of Carbonic Acid. L. T. Bowser. (*J. Ind. Eng. Chem.*, 1912, 4, 203-205.)—The carbonate (1 grm.) is weighed out into the 100 c.c. flask, *F*, and covered with about 50 c.c. of water. The absorbing tower, *T*, containing glass beads, is charged with 10 c.c. of 50 per cent. caustic potash. Sufficient dilute hydrochloric acid to decompose the carbonate is admitted to the flask through the tap-funnel, *S*, the top of which is constricted to allow of connection to a rubber tube, by means of which the acid can be blown in against the back-pressure of the absorbing apparatus. When action slackens, the contents of the flask are heated to boiling, and water is distilled over until the tower is nearly filled. The tap of the funnel is then opened, the absorbing apparatus disconnected at *a*, and its contents discharged through *a* into a 100 c.c. flask by blowing at *b*. A guard tube containing soda lime may be used between the mouth and *b*, but is said to be unnecessary. The tower is filled up with water three times after removing the cork through which *b* passes, and the washings blown over into the 100 c.c. flask. This is then filled to the mark, and 25 c.c. taken for titration. With phenolphthaleïn as indicator, and in presence of 10 c.c. alcohol, normal acid is run in nearly to neutralisation, the titration being completed with $\frac{N}{10}$ acid. The addition of alcohol



facilitates the determination of the end-point. At this stage, the excess of caustic alkali is neutralised and the carbon dioxide all exists as bicarbonate. Methyl orange is then added, and the titration completed as usual, each c.c. of $\frac{N}{10}$ acid required at this stage corresponding to 4·4 mgrm. carbon dioxide in the 25 c.c. or other aliquot taken for titration. Correction must be made for any carbon dioxide in the reagents employed, and this correction is determined by a blank experiment. Where a large number of determinations of carbon dioxide have to be made, the method is more rapid than any other, since three determinations may be completed in an hour, and the results, in the hands of an experienced operator, are accurate to within one drop of an $\frac{N}{10}$ solution. G. C. J.

Analysis of Cement and Cement Raw Materials. B. Blount. (*Institute of Chemistry Lectures on Cement*, 1912.)—*For silica and chief bases:* 0·5 grm. of the cement is thoroughly mixed with a few c.c. of water in a shallow 6 inches evaporating basin; about 20 c.c. strong hydrochloric acid are then added, the whole again well mixed until the cement has dissolved almost completely, evaporated to dryness, and baked for an hour at a temperature of at least 200° C. The dry residue is digested with 30 c.c. strong hydrochloric acid, the solution diluted with water, and the silica and insoluble residue filtered off. The filtrate is precipitated with a slight excess of ammonia and bromine, and heated until only just alkaline. To the filtrate from the alumina and ferric hydroxide about 10 c.c. ammonia (sp. gr. 0·880) are added, the solution heated to boiling, and the lime precipitated by the addition of 25 c.c. of a hot 4 per cent. ammonium oxalate solution. The precipitated calcium oxalate is boiled for two or three minutes, allowed to stand for an hour, and then filtered. The filtrate is evaporated until a pasty residue is obtained, about 50 c.c. strong nitric acid added, the dish covered, and the heating continued until the ammonium salts have been destroyed. The residue is dissolved in a few c.c. of water and hydrochloric acid, heated with a slight excess of ammonia and a few drops of ammonium oxalate solution, and the small precipitate thus obtained is filtered off. This precipitate does not usually exceed 0·0015 grm., and in ordinary work may be neglected. To the filtrate about one-third of its volume of strong ammonia is added, and magnesia precipitated by the addition of 2 to 3 c.c. of a 10 per cent. solution of sodium phosphate. The precipitate is allowed to stand, with occasional shaking, for two hours, filtered, and ignited at the mouth of the muffle. The precipitates of silica and insoluble residue, iron and alumina, and lime are gently burnt off with their filter-papers, and then ignited for an hour at the highest temperature of the muffle.

For insoluble residue and sulphuric anhydride: 0·5 grm. of the cement is dissolved as above, evaporated to dryness, the residue taken up in 10 c.c. hydrochloric acid, and the solution filtered off after dilution. In the filtrate SO_3 is precipitated with barium chloride and estimated as usual. The precipitate of silica and insoluble residue is washed back into the dish and digested with a saturated solution of sodium carbonate, when the insoluble residue only is left undissolved. This is filtered off, washed, ignited, and weighed. Its weight, deducted from that of the silica plus insoluble residue obtained above, gives the quantity of combined effective silica.

For loss on ignition; carbonic acid and water: 0·5 grm. of the cement is ignited in platinum for half an hour in a gas muffle at a temperature of about 800° C.

For carbonic acid: 2 to 5 grms. are decomposed with hydrochloric acid, and the carbonic acid, after purification, is collected in a weighed potash bulb.

For alkalis: 1 grm. of the cement is taken and the silica and chief bases are removed as above. The residue remaining after the destruction of ammonium salts is treated with baryta to remove magnesia and afterwards with ammonium carbonate. The alkalis are weighed as chlorides and separated by means of platinic chloride.

For sulphides: The cement is decomposed with hydrochloric acid in an atmosphere of coal gas, the evolved sulphuretted hydrogen being absorbed in lead acetate solution and weighed as lead sulphate.

The determinations of carbonic acid, alkalis, and sulphides are usually unnecessary.

Separation of alumina and ferric oxide: The freshly precipitated hydroxides obtained either by fusing the ignited oxides with potassium bisulphate, dissolving in dilute hydrochloric acid and precipitating with ammonia, or from a separate portion of cement, are dissolved in dilute hydrochloric acid, evaporated to a small bulk and poured into a strong solution of pure caustic soda. After digestion the precipitated ferric hydroxide is filtered, redissolved in hydrochloric acid and reprecipitated with ammonia.

A more rapid determination may be made by dissolving 1 grm. of cement in dilute hydrochloric acid, reducing with pure granulated zinc and titrating the largely diluted ferrous solution with $\frac{N}{20}$ potassium permanganate solution.

Limestones: If nearly pure, may be analysed as described under cement. The insoluble siliceous matter, after ignition, is treated with hydrofluoric acid and sulphuric acid and ignited: the residue, if appreciable, is fused with sodium carbonate and analysed in the usual manner.

For siliceous and argillaceous limestones which contain a considerable amount of insoluble matter, it is advisable to ignite 1 grm. of the finely-ground sample at the full temperature of the muffle for an hour. The ignited product should be almost completely soluble and may be analysed as described under cement.

Separate portions are taken for the determination of sulphate and carbonic acid in the usual manner.

If necessary, pyrites is estimated by digesting the finely-ground sample with *aqua regia,* removing nitric acid by evaporation with hydrochloric acid and precipitating the total sulphates in the diluted filtered solution by means of barium chloride.

Clay, shale, etc.: 0·5 grm. of the finely-ground sample is fused with about 3 grms. pure sodium carbonate in a platinum crucible for half an hour with occasional shaking. The fused mass is dissolved in hot water and hydrochloric acid and the solution evaporated to dryness and baked for the estimation of silica as described under cement. The filtrate should be evaporated and baked a second time to separate a further small quantity of silica. From this point the analysis is conducted exactly as described under cement. Double precipitation is made if a large proportion of magnesia is present. The purity of the silica is tested by means of hydrofluoric and sulphuric acids. Alumina and ferric oxide are separated by fusion with potassium bisulphate as described above. For rapid work the bisulphate melt may be dissolved in dilute hydrochloric acid, reduced with zinc and titrated with permanganate solution.

The estimation of "loss on ignition," sulphates, carbonic acid and pyrites are made in the usual manner.

If necessary, alkalis may be determined by decomposing the finely-ground sample with hydrofluoric and sulphuric acids. After removing excess of sulphuric acid at a gentle heat, the residue is dissolved in hydrochloric acid, the chief bases removed as usual, and the estimation completed as described under cement.

For shales and clays which contain a large proportion of calcareous matter, it is advisable to extract the sample with hydrochloric acid and to analyse the soluble and insoluble portions separately. G. J. A.

Quantitative Separation of Copper from Arsenic, Aluminium, Zinc, Tungsten, and Tin by Means of Hydrogen Peroxide in Presence of Sodium Hydroxide and Sugar. P. Jannasch and O. Routala. (*Ber.*, 1912, **45**, 598-604.) —When a solution of copper sulphate is mixed with sufficient cane sugar, so that no precipitate is formed on the addition of excess of sodium hydroxide, the whole of the copper may be precipitated as cuprous oxide by the addition of hydrogen peroxide and warming. For the estimation of copper alone, 0·3 grm. of copper sulphate is dissolved in about 75 c.c. of water and 3 grms. of cane sugar and 10 c.c. of a 10 per cent. solution of sodium hydroxide are added in the cold; 20 to 25 c.c. of 6 to 8 per cent. hydrogen peroxide are then added and precipitation is completed by warming for about fifteen minutes on the boiling water-bath. The cuprous oxide is filtered off, washed with boiling water, and incinerated until fully oxidised. For the separation of copper from other metals it is generally advisable to have about 6 grms. of sodium hydroxide and 5 grms. of cane sugar present in 100 c.c. of the solution. The complete precipitation of the copper may be controlled by adding a further 5 c.c. of the peroxide after heating on the water-bath. After the cuprous oxide has been filtered off, the sugar must be removed before the other metals can be precipitated, except in the case of arsenic. Arsenic is precipitated as magnesium ammonium arsenate, aluminium by means of ammonia, zinc as carbonate, and tin as stannic acid. For the oxidation of the sugar, the liquid should be evaporated in a platinum dish, treated several times with strong nitric acid, evaporating to dryness each time, then with nitric acid and hydrogen peroxide, and finally heated so as to fuse the contents of the dish. Precautions are necessary to prevent the oxidation of the cuprous oxide during filtration. J. F. B.

Valuation of Fluorspar. E. Bidtel. (*J. Ind. Eng. Chem.*, 1912, **4**, 201.)— The following method gives excellent results when applied to Illinois fluorspars, which contain, in addition to calcium fluoride, silica (0·4 to 13 per cent.), and calcium carbonate (0·2 to 15 per cent.), and small amounts of lead sulphide (nil to

2 per cent.), and ferruginous zinc sulphide (nil to 2 per cent.). One grm. of the dry and finely-powdered sample contained in a small Erlenmeyer flask is digested with 10 c.c. of 10 per cent. acetic acid on the water-bath for an hour, with occasional shaking. The undissolved matter is filtered off on a 7 cm. filter, and washed four times with hot water. Under these conditions ·0015 grm. of calcium fluoride passes into the filtrate. The filter-paper is burnt and the residue ignited and weighed. The loss in weight due to treatment with acetic acid, less ·0015 grm., is calculated as calcium carbonate. In some fluorspars other carbonates may be present, but their amount is usually very small. About 1 grm. of yellow mercuric oxide in the form of a cream is next mixed in with the residue in the platinum crucible, and the mixture dried, raised to a dull red heat, cooled, and weighed. Any lead originally present as sulphide will now exist as sulphate. About 2 c.c. of hydrofluoric acid are next added to expel silica, and, after evaporation to dryness, the operation is repeated twice with about 1 c.c. of hydrofluoric acid. A few drops of hydrofluoric acid are again added, together with some macerated filter-paper, and this is followed by a few drops of ammonia to precipitate the iron. The contents of the crucible are next dried, raised to a dull red heat, cooled, and weighed. The loss since the last weighing is reported as silica. After addition of 2 c.c. of hydrofluoric acid and a few drops of nitric acid, the crucible is covered and its contents digested on the water-bath for half an hour. The lid is then removed, and the contents of the crucible are evaporated to dryness. If not perfectly white, the residue is evaporated again with hydrofluoric acid. After moistening with a few drops of hydrofluoric acid, the residue is digested for half an hour on the water-bath with 10 c.c. of a solution containing 400 grms. ammonium acetate, 20 grms. citric acid, and 200 c.c. of strong ammonia per litre. The insoluble matter is filtered off, washed with ammonium acetate solution, and then with hot water, ignited, and weighed. In the author's experience, the residue thus obtained is pure calcium fluoride, and ·0029 grm. should be added to its weight for losses due to the solubility of calcium fluoride in acetic acid and in ammonium acetate. The residue should, however, be examined for barium by conversion to sulphate, fusing the sulphate with sodium carbonate and solution of the fused product in hydrochloric acid. Illinois fluorspars do not contain heavy spar or gypsum, but the frequent request to analysts to estimate barium sulphate leads the author to suppose that it is a not uncommon constituent of fluorspar. It is pointed out that calcium sulphate, if present, would be partly leached out by the dilute acetic acid and estimated as carbonate.

G. C. J.

Estimation of Magnesia in Admixture with Magnesium Carbonate. V. Fortini. (*Chem. Zeit.*, 1912, 36, 270-271.)—Where the import duty on magnesia is high the attempt is sometimes made to smuggle it in admixed with magnesium carbonate, and under the description of magnesium carbonate, on which the duty is never more than half as much and may be less. A determination of carbon dioxide alone occupies some time, and alone is insufficient, as the description of the article may allow the presence of asbestos and other substances which would reduce the percentage of carbon dioxide below that corresponding to $MgCO_3$. Advantage may be taken of the fact that magnesia dissolves in hydrochloric acid with considerable evolution of heat, whereas this is not the case with magnesium carbonate. The author makes use of Tortelli's thermoleometer (Analyst, 1909, **34**, 168), but any calorimeter which allows of the use of hydrochloric acid of sp. gr. 1·1 may be used. With Tortelli's instrument, 1 grm. of magnesia added to 25 c.c. of dilute hydrochloric acid raises the temperature of the acid 37° C., 0·1 grm. raising it 3·7° C. Commercial magnesium carbonate always contains basic carbonate, but never enough to vitiate this test, which aims at detecting gross frauds such as alone could benefit the importer.

G. C. J.

Estimation of Nitrates and Nitrites in Water by Schlœsing's Method. A. Huizinga. (*Zeitsch. anal. Chem.*, 1912, **51**, 292.)—This method does not always yield correct results when the water is evaporated after the addition of magnesia, and the gas, obtained by boiling the residue with hydrochloric acid and ferrous chloride, treated with sodium hydroxide solution. The results are invariably inaccurate when nitrites are also present in the water. The following modification of the method of preparing the water total solids for the estimation is therefore recommended: One litre of the water (in the case of rain-water 5 litres are taken) is rendered alkaline with potassium hydroxide and evaporated to a volume of about 100 c.c.; 4 c.c. of $\frac{N}{10}$ permanganate solution are then added, the mixture is boiled, filtered, and the filtrate again boiled after the addition of a further quantity of permanganate solution and 2 c.c. or more of dilute sulphuric acid. The acid solution is now rendered alkaline, filtered, and the filtrate evaporated to a volume of about 20 c.c.; 2 c.c. of acetic acid, or a quantity sufficient to acidify the solution, are added, and the evaporation is continued to dryness. The residue is then treated in a Schlœsing's apparatus in the usual way, but, carbonates having been decomposed by the evaporation with acetic acid, the evolved gas is not collected over sodium hydroxide solution. The results obtained do not differ by more than 0·1 mgrm. from the quantity of nitrogen actually present.

W. P. S.

Use of a Mixture of Nitric Acid and Hydrogen Peroxide in Analytical Work. P. Jannasch. (*Ber.*, 1912, **45**, 605-607.)—1. *For the Destruction of Organic Matter.*—In forensic analysis, after the animal matter has been destroyed by treatment with potassium chlorate and hydrochloric acid, the search for metals of the hydrogen sulphide group is complicated by the formation of yellowish-brown precipitates, mainly of organic nature. This organic matter is not readily destroyed by repeated evaporation with fuming nitric acid, but it is easily oxidised by a mixture of nitric acid and hydrogen peroxide. The precipitate obtained with hydrogen sulphide is treated once or twice with red fuming nitric acid and heated to dryness on the water-bath. The residue is then treated in a similar manner with a mixture of ordinary 65 per cent. nitric acid, sp. gr. 1·4, and 15-20 per cent. hydrogen peroxide obtained by diluting Merck's 30 per cent. "perhydrol" with water. In this way the metals are completely freed from organic impurities. This method possesses the advantage of introducing no non-volatile reagents.

2. *For the Removal of Ammonium Salts.*—The ordinary 65 per cent. nitric acid, sp. gr. 1·4, is recommended in preference to the fuming acid for the purpose of

removing ammonium chloride by evaporation. In the case of ammonium nitrate a little concentrated hydrochloric acid is added at the same time. Ammonium sulphate cannot be volatilised by nitric acid alone; repeated evaporation with strong hydrochloric and nitric acids is recommended, or else the mixture of nitric acid and hydrogen peroxide. Ammonia is driven off from ammonium phosphate best by evaporation with strong nitric acid and bromine. In the analysis of complex silicates, after the precipitation of the calcium as oxalate, the filtrate is evaporated nearly to dryness, the residue is moistened with water to a pasty consistence and then evaporated to dryness with 25 c.c. of 65 per cent. nitric acid. If this operation be repeated with 10-15 c.c. of the acid, only traces of ammonium salts remain, which can readily be volatilised by short heating on an asbestos-covered gauze. The magnesia is then precipitated by mercuric oxide. J. F. B.

New Method for Testing Paint Films and Preservative Coatings for Iron and Steel. W. C. Slade. (*J. Ind. Eng. Chem.*, 1912, 4, 189-198.)—The method depends on the periodical measurement of the electric current passing through a circuit which includes a bare wire anode, a normal solution of potassium chloride, a cathode consisting of a wire helix of specified dimensions, coated with the paint to be investigated, and an external resistance of 100 ohms. The apparatus illustrated in the paper shows several electrolyte tanks about 28 ins. by 7 ins. by 8 ins. each of which will accommodate 14 of these elements or "paint cells," as they are termed. The electrolyte in the neighbourhood of the painted helix is continuously aerated, whilst that surrounding the bare wire is protected from access of oxygen. A clockwork arrangement provides for all the paint cells being elevated out of the electrolyte at 3, 6, 9, and 12 o'clock, and for their re-immersion at 1, 4, 7, and 10 o'clock. At or about 2, 5, 8, and 11 o'clock, the current in each circuit is measured after cutting out the external resistance and substituting a milliammeter, also of 100 ohms resistance. Most of the author's experiments have extended over 300 hours, but it appears that as much information may be gained in 200 or perhaps even in 150 hours. Apparatus has been designed and is illustrated in the paper for securing paint films of uniform thickness. The helices are dipped in the well-shaken paint mixture, allowed to drain until dropping begins, and then mounted horizontally on a slowly rotating wheel, so that the paint flows round the wire instead of draining away in drops. If the electrolytic theory of corrosion be admitted, the method reproduces closely the conditions to which painted steel work is exposed. When a paint-cell is first connected up, no current, or a current of a few thousandths of a milliampère, passes. But with time this current always increases and may reach 0·5 milliampère or thereabouts. This is explained as follows: When the electrolyte has penetrated the paint-film sufficiently to establish contact with the iron, the hydrogen ions that tend to polarise are depolarised by the oxygen of the air which is in solution, and the operation of the cell commences. With increased porosity comes an increased rate of diffusion, and an increased rate of depolarisation, and a decrease in film resistance. The current consequently increases. At this time any depolarising power possessed by linoxyn that may still be unsaturated should show its influence. To maintain the equilibrium of ionic charges within the solution, metallic iron must pass into solution at some point in the form of ferrous ions. This takes place almost wholly at the undepolarised electrode. The ferrous ions unite with the hydroxyl ions (corresponding to the hydrogen ions removed by depolarisation) to form ferrous hydroxide, which in turn is oxidised to ferric hydroxide. Some rust forms at the cathode, but its amount is very small compared with that formed at the anode. The numerical results obtained admit of varied mathematical treatment in attempts to classify paints as a result of this test. Several such methods of treatment are discussed. G. C. J.

Estimation of Phosphoric Acid in Presence of Colloidal Silicic Acid. P. Mélikoff and M. Becaia. (*Comptes rend.*, 1912, **154**, 775.)—The usual method of estimating phosphoric acid is faulty if colloidal silicic acid is present, but accurate results may be obtained by the use of permolybdate reagent (Analyst, 1912, 107). Thus, two portions of 50 c.c. disodium phosphate solution, to each of which had been added 50 c.c. colloidal silicic acid containing 0·581 grm. SiO_2, gave 0·1725 and 0·2067 instead of 0·1211 grm. P_2O_5, on estimation, by means of magnesia mixture, of the phosphoric acid in the precipitate obtained with molybdate, which is thus shown to carry down silica. When, however, this precipitate of mixed phosphomolybdate and silicomolybdate is treated four times in twenty-four hours with permolybdate reagent (equal volumes of 30 per cent. hydrogen peroxide and 15 per cent. ammonium nitromolybdate) and washed on a filter with the reagent, the filtrate heated to 40° or 50° C. to destroy excess of hydrogen peroxide, and the phosphoric acid determined in it by means of magnesia mixture, 0·1193 and 0·1209 grm. P_2O_5 (instead of 0·1211) were obtained. O. E. M.

Estimation of Small Quantities of Potassium. E. A. Mitscherlich, K. Celichowski, and H. Fischer. (*Landw. Vers.-Stat.*, 1912, **76**, 139-155; through *Chem. Zentralbl.*, 1912, I., 853.)—The method proposed consists in precipitating the potassium in the form of potassium cobaltinitrite and titrating this nitrite with permanganate solution; it is suitable for the analysis of soil extracts, and is stated to be more accurate than the platinum method. Two hundred and fifty c.c. of an aqueous extract of a soil are treated with 5 c.c. of concentrated nitric acid and 5 drops of dilute sulphuric acid, and evaporated to dryness; after the residue has been ignited in order to destroy organic matter, it is treated with 1 drop of concentrated sodium carbonate solution and a few c.c. of water, and again evaporated to dryness. The residue is now heated until fusion commences, cooled and neutralised with nitric acid; any excess of acid is removed by evaporating the mixture to dryness. Five c.c. of hot water are added to the residue, and the solution is treated with 3 c.c. of a 10 per cent. cobalt chloride solution and 5 c.c. of a 10 per cent. sodium nitrite solution; the mixture is now evaporated to dryness at a temperature of 80° to 90° C., and stirred frequently during the evaporation. When cold, the residue is treated with 3 c.c. of 10 per cent. acetic acid, 10 c.c. of water are then added, and the insoluble portion is collected on a filter and washed six times with a 2·5 per cent. sodium sulphate solution, 3 c.c. of this solution being used each time. A definite volume of $\frac{N}{50}$ potassium permanganate solution (in quantity somewhat more than sufficient to oxidise the nitrite) is placed in a beaker, diluted with 100 c.c. of water, and heated nearly to boiling on a water-bath; the contents of the filter are now introduced into the beaker, and when manganese dioxide begins to separate, about 2·5 c.c. of dilute sulphuric acid are added drop by drop. As soon as the yellow nitrite precipitate has dissolved, the excess of permanganate is titrated with $\frac{N}{10}$ oxalic acid solution. Each c.c. of $\frac{N}{10}$ permanganate solution is equivalent to 0·000157 grm. of K_2O (*cf.* ANALYST, 1911, **36**, 606). W. P. S.

Iodometric Estimation of Selenium in Sulphur and Pyrites. P. Klason and H. Mellquist. (*Zeitsch. angew. Chem.*, 1912, **25**, 514-518.)—Sulphur (100 grms. or more) is placed in porcelain boats, each of which contain 10 to 15 grms, in a combustion tube 100 cm. in length and 20 mm. in diameter. At the forward end of the tube is an asbestos plug which is kept cool. A current of air is drawn through the tube and the combustion of the sulphur started by means of a Bunsen burner. The action then proceeds without external heating, but at end of the combustion each boat is heated to volatilise any adherent selenium before it is withdrawn. The results show that the cool asbestos plug retains all but an unweighable trace of the selenium which passes forward as a fog with the small quantity of sulphur trioxide which is always formed. The radish-like odour characteristic of selenium compounds may always be detected, but the results show that the loss at this stage must be negligible, and that under the conditions of the experiment the volatile selenium suboxide of Berzelius can only be formed in traces. The tube and asbestos plug are treated with a solution of potassium cyanide to dissolve the selenium, and the resulting solution made strongly acid with hydrochloric acid and evaporated until crystallisation begins, in order to precipitate the whole of the selenium as selenious acid. The precipitate is collected on a Soxhlet asbestos filter of large dimensions, with the wide portion of the tube 280 mm. long and 10 mm. in diameter. The filtering plug of asbestos is supported on a platinum cone, and, after drying the precipitate, a second plug of asbestos is introduced an inch or so into the tube. The precipitate is next heated and a current of oxygen led through the tube. Since the precipitate invariably contains some sulphur, and as selenious acid is reduced by sulphur dioxide, the oxidation of the selenium to selenious acid does not begin until all the sulphur has been expelled. The selenious acid sublimes to a cooler part of the tube, and, if contaminated by unoxidised selenium, the flame is directed on the sublimate, which is driven backwards and forwards along the tube until quite white. The selenious acid is finally dissolved in water and the solution diluted to 100-300 c.c., and acidified with two or three drops of concentrated hydrochloric acid for each 100 c.c. of solution. The acidified solution is heated in a flask on the water-bath, the air in the flask displaced by carbon dioxide, and 2 to 5 grms. potassium iodide added. When the iodide has dissolved, the flask is shaken, cooled in running water, and placed in the dark for an hour. The liberated iodine is then titrated with $\frac{N}{100}$ thiosulphate, using starch as indicator. $SeO_2 = 4I$. Selenium when present in sulphur in the proportion of 0·5 oz. to the ton, or less, may be estimated in this way with an error not exceeding 5 per cent., but, when determining such small quantities, the hydrochloric acid and potassium iodide used must be free from traces of chlorine and iodate respectively.

Pyrites, when heated in boats in a combustion-tube even in a current of oxygen, does not part with the whole of its selenium, about 55 per cent. remaining with the ash if the heating be by means of a Bunsen burner. But the importance of this work depends on the recent observation (Klason, *Verein. Zellstoff u. Papierchemiker*, 1909, 61) that minute traces of selenium in the liquor of a sulphite-cellulose factory exercise a most prejudicial influence, and in factory practice much of the selenium present in the pyrites remains with the ashes. The authors, therefore, consider that the result of a roasting experiment gives, perhaps, more useful practical information than an estimation of total selenium, though they practise both. When a Bunsen flame is used, the ashes contain about 8 per cent. of sulphur, whereas the burned pyrites of a sulphite cellulose factory is said to contain 4 per cent. of sulphur, as a rule. The authors, therefore, complete their combustion in an electric furnace at such a temperature that the ashes contain about 4 per cent. of sulphur if the heating is discontinued when the odour of sulphur dioxide can no longer be detected. Under such conditions as much as 75 per cent. of the selenium may be expelled from the pyrites.

For the estimation of total selenium in pyrites, 25 grms. are dissolved in concentrated hydrochloric acid, with addition of potassium chlorate. After filtering from insoluble matter and reducing iron to the ferrous state by means of zinc, the selenium is precipitated by addition of stannous chloride to the hot solution. The elementary selenium is filtered off on asbestos, and freed from arsenic by solution in potassium cyanide and reprecipitation by means of hydrochloric acid. The pure selenium is then collected on the Soxhlet filter, and oxidised to selenious acid, and estimated volumetrically as already described. G. C. J.

NOTE BY ABSTRACTOR.—The objection to selenium in bisulphite lyes intended for use in sulphite cellulose factories depends on the fact that even a small fraction of 1 mgrm. per litre brings about the rapid destruction of the lye by production of sulphuric acid and gypsum. Selenium is 500 times more powerful than sulphur as a catalytic agent in the production of sulphuric acid from sulphurous acid.

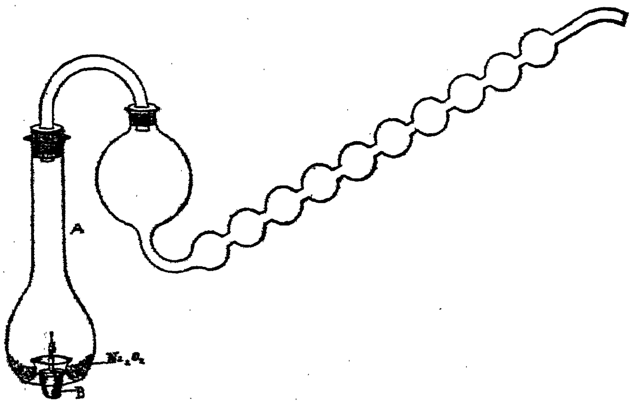
Estimation of Sulphur and of Halogens in Small Quantities of Organic Substances. J. Donau. (*Monatsh.*, 1912, **33**, 169-176.)—The method employed is essentially that of Carius, but the apparatus described has been devised so as to admit of the application of that method to quantities such as are weighed on a micro-balance. The tubes employed are about 9 cm. long before sealing off, with a thickness of wall of 1 mm. and an internal diameter of 6 mm. The end first sealed up is given a just perceptibly bulbous form, whilst the next centimetre of the tube is blown out into a distinct but still very small bulb. The substance for analysis is weighed out in and handled by means of a special platinum spoon, which is constructed from a disc of foil, 3 mm. in diameter and 0·05 mm. thick, and a piece of wire 3 cm. long and 0·1 mm. in diameter. The disc is given a hemispherical shape by placing it on a rubber bung and pressing with a suitably-shaped piece of wood. The wire is then welded on to the cup thus formed. Into this spoon not more than 5 mgrms. of the substance are placed, and the spoon, resting in a larger piece of foil, is then weighed on the micro-balance. By means of a special pair of forceps the spoon is pushed right to the end of the Carius tube, held in a horizontal position, and its contents are transferred to the more distant bulb by turning the handle of the spoon through 180 degrees. The spoon is then withdrawn, replaced on the foil on the micro-balance, and reweighed. By means of a similar spoon, silver nitrate, or barium chloride, as the case may be, is introduced into the more distant bulb, and then 2 drops of nitric acid are introduced into the second bulb by means of a pipette with a bent point. Still keeping the tube horizontal, it is sealed off so as to be about 7 cm. long. The terminal capillary need not be longer than 0·5 cm. The tube is now placed in a vertical position, capillary uppermost, in a furnace of special construction. This consists of a cylinder or prism of copper some 10 cm. high, with a cross-sectional area of 30 to 40 sq. cm. From the top of this cylinder or prism, a number (*e.g.*, 4) of 16-mm. holes are bored to within 1 or 2 cm. of the bottom. One of these holes accommodates a thermometer; the others form the chambers for the reception of the Carius tubes, which rest on asbestos plugs. The open mouths of the chambers are covered with an asbestos plate, and the furnace is placed on a tripod and heated by means of a powerful Bunsen burner. When decomposition is complete and the tubes have cooled, the internal pressure is released in the usual manner; but the tubes are sprung in the plane of maximum diameter of the upper bulb which contained the nitric acid before the tubes were placed in a vertical position after sealing. The end thus cut off, which may be given a lip, provides a minute beaker for handling the precipitate before filtering. The washings from the larger half are transferred to this. Precipitates of barium sulphate are evaporated several times with hydrochloric acid, the residue taken up in water containing hydrochloric acid, and the precipitate filtered off on the micro-filter previously described (ANALYST, 1912, 68). It is washed with about 5 drops of hot water, dried, placed on a piece of platinum-foil and ignited, placed quickly whilst still hot in a desiccator, removed after half a minute, and weighed. In halogen determinations, the water used for washing the rejected portion of the Carius tube is evaporated to 1 or 2 drops, a few drops of water are added, and the silver halide filtered and washed as already described. As superficial reduction of the silver halide is unavoidable, the platinum filter is exposed for about a minute to an atmosphere of chlorine or bromine, as the case may be, and then dried with its contents at 130° C. Experiment shows that the platinum filter is not sensibly attacked by exposure in this way to chlorine or bromine, and the treatment must not be omitted, or low results will be obtained. One mgrm. of sulphur, chlorine, or bromine, can be estimated with an error not exceeding 2 per cent.

G. C. J.

Estimation of Sulphur in Petroleum. J. McC. Sanders. (*J. Chem. Soc.*, 1912, 101, 358-365.)—Two methods are given for the estimation of the total sulphur of kerosenes, and a method for loosely-combined sulphur, all of which have yielded rapid and good results with all classes of petroleum.

For Total Sulphur—(1) *With the Parr Calorimetric Bomb.*—From 2 to 3 grms. of sample in a porcelain basin are treated with 0·01 grm. of potassium bromide dusted over the surface, and 4 c.c. of fuming nitric acid run in from a pipette. When action has ceased, the liquid is evaporated at 100° C., till somewhat viscous and dark brown in colour. About 0·5 grm. light magnesium oxide is now stirred into the mass, which is heated over a small flame, with continuous stirring, till the product tends to solidify on cooling. The amounts of magnesia used and the time of heating should be such as to allow the easy removal of the product on cooling, the remaining trace of oily matter being taken up with a little magnesia, warming, and scraping out with a spatula. The granular mass resulting is placed in the calorimeter, the standard amount of peroxide added, and the mixture stirred with an iron rod. The bomb is closed and fired with due precautions by the electric fuse. When the combustion is over (forty-five seconds), the bomb is cooled, opened, and the fused mass dropped into 25 c.c. water in a nickel beaker, the washings from the bomb added, and the liquid cautiously acidified with hydrochloric acid. After quick filtration through cotton-wool the liquid is boiled to get rid of chlorine, neutralised with ammonia, again acidified with hydrochloric acid, and the sulphate precipitated with barium chloride as usual. The results agree closely with those given by the Mahler bomb. When the amount of sulphur present is less than 0·01 per cent., Method 2 is more accurate.

(2) *Modified Lamp Method.*—A special form of lamp-chimney and lamp are required; the chimney is made from a Kjeldahl Jena-glass flask by softening the bottom in the flame and producing a re-entrant bulb by aspiration (see figure). The annular space round the bottom of this chimney holds 6 grms. of sodium peroxide and 0·5 grm. of anhydrous cobalt chloride, and this apparatus is connected with a Meyer absorption-tube, the corks being covered with tin-foil. Three or four bulbs are filled with water to which a little sodium peroxide has been added. The lamp consists of a very small platinum crucible closely packed with recently ignited asbestos, in which a small wick of asbestos is embedded. The wick passes through the central perforation of a Rose's crucible lid, and is supported by a small piece of pipe-clay tubing of such length that when the lamp is in position the top of the wick support is a little above the upper rim of the annular cavity of the chimney. Four to five grms. of the sample are weighed into the lamp, which is placed in position and lighted, the filter-pump being so operated as to draw a rapid current of air through the apparatus. When nearly all the petroleum has been burnt, the bottom of the lamp is heated by a small spirit-flame to drive off the remaining petroleum, the lamp being finally heated to low redness. After cooling, the chimney and liquid in the



bulbs, etc., are washed into a large beaker, the liquid acidified by hydrochloric acid, boiled to expel chlorine, and precipitated by barium chloride in the usual manner.

Method for Loosely-Combined Sulphur.—Some commercial samples of lamp oils contain sulphur in the form of unstable compounds, which may be estimated as follows: Finely-cut copper turnings are warmed with potassium cyanide solution (10 per cent.) till the metal is cleaned, when it is washed with air-free water, and finally with methyl alcohol, and the copper closely packed into Erlenmeyer flasks (100 c.c.) so as to fill them. Fifty c.c. of the petroleum are run into such a flask, which is closed with a cork carrying an air-condenser tube. The flask is heated for ten minutes on a hot-plate, the cork and tube removed, and the petroleum evaporated till only 10 c.c. remain. After cooling, 50 c.c. potassium cyanide solution are added, the liquid warmed in contact with all the turnings, which are stirred to facilitate the solution of the sulphide. The solution is poured through cotton-wool, the metal washed with water, and this transferred to the wool filter. From this solution the sulphur may be obtained by oxidation with bromine, and treatment with hydrochloric acid and barium chloride as usual.

When the density of the sample is known at the room-temperature, it is convenient to measure, instead of weigh, the amount required, the sulphur per cent. being found as follows when 3 c.c. are taken for analysis:

$$S = \frac{P \times 45{\cdot}76}{D \times 10};$$

where S = percentage of sulphur, P = weight of barium sulphate found, and D = density of the sample. A. R. T.

New Method for the Separation of Thorium. T. V. Smith and C. James. (*J. Amer. Chem. Soc.*, 1912, 34, 281-284.)—Sebacic acid in neutral solutions of thorium salts gives a voluminous granular and readily filtered precipitate, which, when dried and ignited, may be weighed as thorium dioxide. The reaction is quantitative, and shows good agreement with the results obtainable with oxalic acid. The precipitant is used in boiling solution saturated at that temperature. The mean of six determinations with sebacic acid showed an average weight of 0·2788 grm. of the dioxide in 50 c.c. of standard thorium nitrate solution as against 0·2786 grm. by the oxalic acid method. Solutions of cerium, praseodymium, lanthanum, neodymium, samarium, and yttrium, give no precipitate even on boiling; dysprosium, erbium, and thulium were not tested, as these elements rarely occur in ordinary thorium minerals. Test mixtures of rare earths were made up with varying quantities of thorium, the thorium in all cases being found with a very close approximation to accuracy. The separation is recommended for use on the large scale, as sebacic acid is readily prepared by heating castor oil soap with sodium hydroxide, when, owing to its slight solubility in cold water, it can be easily recovered. Other acids which give no precipitate with the rare earths, but precipitate thorium more or less quantitatively, were also investigated, but, owing to difficulties of filtration, or for other reasons, were not found satisfactory. Among them were phenoxyacetic, mucic, anisic, and aspartic acids. Pyrotartaric acid appears to precipitate thorium quantitatively in hot solutions, but is more expensive than sebacic acid. H. F. E. H.

Rapid and Exact Estimation of Tungsten in Ferrotungsten. R. Fieber. (*Chem. Zeit.*, 1912, **36**, 334.)—The material (0·5 grm.) is reduced to the finest possible state of division and oxidised by warming gently with 10 to 15 c.c. of bromine. Concentrated hydrochloric acid (30 c.c.) is next added, and, if anything remains undissolved, a further 5 to 10 c.c. of bromine. The excess of bromine is driven off by heating on the hot-plate, and then 1 to 2 c.c. of nitric acid and 2 or 3 drops of hydrofluoric acid are added. The object of the latter is to prevent the adherence of tungstic acid to the beaker during the subsequent evaporation. The contents of the beaker are next concentrated to very small bulk, the residue taken up with 40 c.c. of dilute (1 : 5) hydrochloric acid, the solution boiled, and the precipitate given some minutes to settle. The precipitate is filtered off on a double filter, washed alternately with hot water and dilute hydrochloric acid, moistened with a solution of ammonium nitrate, burnt, ignited, and weighed. The tungstic acid thus obtained is pure yellow in colour, and contains, as sole impurity, silica, which is removed by treatment with hydrofluoric and sulphuric acids. The whole series of operations may be performed in two to two and a half hours, and the results are said to be exact. G. C. J.

Analysis of Ferro-zircon. L. Weiss and W. Trautmann. (*Zeitsch. anal. Chem.*, 1912, **51**, 303-304.)—The method of separating zirconium oxide from iron oxide, alumina, and silica, described by Wunder and Jeannert (ANALYST, 1912, 36), is found to be quite untrustworthy. When zirconium oxide is fused with sodium carbonate and the melt extracted with water, the insoluble residue contains a considerable quantity of sodium zirconate which is readily dissolved by treatment with hydrochloric acid; the quantity of zirconium oxide thus passing into solution amounts to from 12 to 14 per cent. of the total quantity, and a further 10 per cent. is rendered soluble by a subsequent fusion. Zirconium oxide is still more readily attacked by sodium carbonate when iron oxide is present. Further, the separation of alumina and silica from zirconium oxide and iron oxide cannot be attained by fusing the mixture with sodium carbonate and extracting with water. Considerable quantities of alumina and silica remain insoluble. W. P. S.

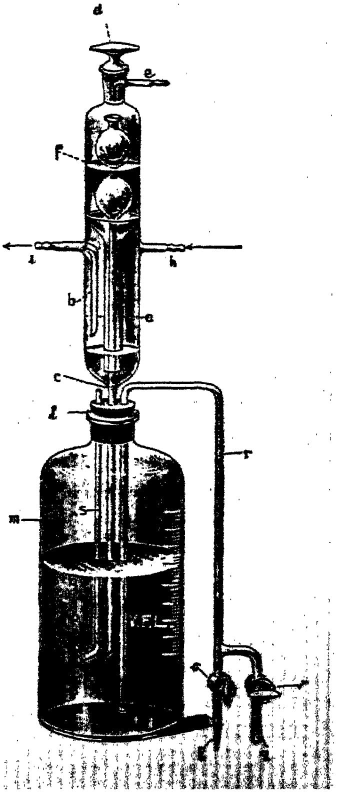
Modification of the Modified Winkler Method for the Determination of Sulphates in Water. H. W. Brubaker. (*J. Amer. Chem. Soc.*, 1912, **34**, 284-285.)—In the modified Winkler process the exact neutralisation of the hydrochloric acid with soda is tedious. The author changes the acidity to acetic acid by adding sodium acetate, and recommends the following procedure: 150 c.c. of the sample is measured into an Erlenmeyer flask, 10 drops of strong hydrochloric acid are added, and 0·1 to 0·2 grm. of barium chromate sprinkled in. The whole is brought to the boil, cooled at once, and then 5 grms. of sodium acetate are added. After filtration, 100 c.c. of the clear filtrate are transferred to a colorimeter cylinder, and made alkaline with sodium hydroxide. The standard for comparison should be made slightly stronger than this solution, and should be made up to 100 c.c. after making alkaline with soda. A blank determination, using distilled water, must be made to determine the correction to be allowed for the solubility of the barium chromate. Results by the gravimetric barium sulphate method, the author's method, and the modified Winkler method, showed an extreme variation of 1·5 parts per million when 100 parts of SO_4 per million were present. H. F. E. H.

APPARATUS, ETC.

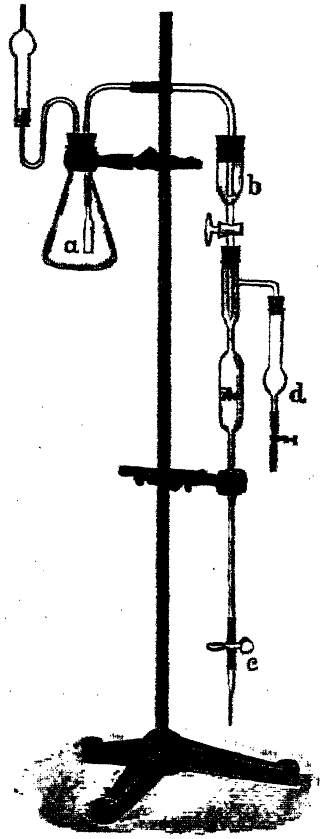
Modified Butyrometer Tube for the Estimation of Fat in Cheese. H. van Gulik. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 99-101.)—The tube described resembles in shape the ordinary form of tube, but is open at both ends, and the graduated portion is divided into forty divisions, which show percentages of fat directly when 3 grms. of cheese are used for the estimation; the ends of the tube are closed with rubber stoppers. For the estimation, 3 grms. of the finely-divided cheese are introduced into the tube, which has been previously one-half filled with sulphuric acid of sp. gr. 1·50. The tube and its contents are then heated in a water-bath at 70° C. until the cheese is dissolved, 1 c.c. of amyl alcohol is next added, and then sulphuric acid, so that the tube is filled up to the 35 mark. After the mixture has been submitted to centrifugal action for four minutes, the volume of the fat is read when the contents of the tube have been brought to a temperature of 65° C.

W. P. S.

Apparatus for Controlling the Evaporation or Distillation of Liquids, and for Cutting off the Supply of Gas to a Burner after any Desired Length of Time. E. Schirm. (*Zeitsch. anal. Chem.*, 1912, **51**, 301-302.)—The supply of gas to a burner below a distillation flask passes through the tubes *h* and *i*, and at the commencement of a distillation the stem *c* is passed through the cork of the flask so that the lower end of the stem is at any desired distance below the surface of the liquid. The cylindrical portion of the apparatus is filled with water up to the level *a*, and on applying suction to the tube *e* this water is drawn up to the level *f*; the tap *d* is then closed. When the distillation has proceeded so far that the level of the solution in the flask falls below the lower end of the stem *c*, air passes upwards into the cylindrical portion of the apparatus, and the level of the water falls, closing the end of the tube *b* and cutting off the supply of gas to the burner. If the apparatus be mounted as shown in the illustration, the supply of gas to a burner may be cut off by regulating the flow of water from the bottle *m* through the jet *t*; the tap *p* serves the purpose of



regulating the level of the water in the bottle. The time taken for the level of the water to sink from one division to another on the scale at the side of the bottle is determined, and the position of the end of the stem *c* may then be so adjusted that after a certain time the level of the water falls below it, allowing air to pass up the stem, and causing the gas-supply to be cut off as described above. W. P. S.



Apparatus for Measuring Liquids in an Atmosphere Free from Carbon Dioxide. T. Ekecrantz and H. Palme. (*Zeitsch. anal. Chem.*, 1912, 51, 292-294.)—The apparatus shown in the illustration is suitable for use in estimating the strength of calcium hydroxide or barium hydroxide solutions, or for measuring off solutions prepared with a volatile solvent. The solution is prepared in the Erlenmeyer flask, which is then attached to the rubber stopper, and a current of air free from carbon dioxide is drawn through the apparatus, the tube *d* being meanwhile closed and the clip *c* opened. The two drying-tubes are filled with soda-lime. The filter-tube *a* is now lowered into the solution contained in the flask, the clip *c* is closed, and suction is applied at *d* so that the solution is drawn over into the pipette. The first 50 c.c. collected in the pipette are run off, and a second quantity of 50 c.c. of the solution drawn over and used for the estimation. While the pipette is being emptied the tap below the portion *b* must be closed. W. P. S.

Use of Magnesia Rods instead of Platinum Wire in Analytical Work. E. Wedekind. (*Ber.*, 1912, 45, 382-384.)—For many purposes in the laboratory rods of magnesia composition may be used instead of platinum wire. These rods are not composed of pure magnesia, but of a more resistant composition, which is employed in the manufacture of supports for incandescent mantles. They are obtainable in lengths of 15 cm. and diameter of 1 mm.; after use the contaminated end of the rod may be broken off. The rods are useful for demonstration purposes, for flame tests, borax beads, manganese and chromium melts, and for driving off volatile matters from small quantities of substance. Spoon-shaped rods of larger diameter are also obtainable. J. F. B.

Constant-Temperature Heating Apparatus for Explosives and Experiments on the Decomposition of Nitrocelluloses. J. S. Brame. (*J. Soc. Chem. Ind.*, 1912, 31, 159.)—The vapour of a liquid of suitable boiling-point (toluene for 110° C.; toluene 3 volumes, xylene 1 volume, for 115° C.; amyl alcohol for 130° C.) is conducted from a copper boiler with safety tube through a copper jacket or heater, and thence through a condenser back to the boiler; it may be shut off from the heater, when it passes directly to the condenser through a ball-valve. The 2-grm. sample, dried at 40° C., is heated *in vacuo* in a 45 c.c. decomposition tube placed in the heater, which contains oil of high flash-point, and the water coming off in the first fifteen minutes is removed by a mercury pump, communication with which is then shut off, and the heating continued for six hours. The gases are then pumped off into a measuring burette. Experiments at 115° and 130° C. on insoluble, soluble, and highly soluble nitrocelluloses at progressive stages of stabilisation, gave results which showed small differences of stability more clearly than the parallel Will tests.

O. E. M.

Application of Ultrafiltration in Analytical Chemistry. R. Zsigmondy, E. Wilke-Dörfurt and A. von Galecki. (*Ber.*, 1912, **45**, 579-582.)—The use of collodion film filters, which has proved so valuable in colloid investigations, may with advantage be extended to the ordinary operations of gravimetric analysis. The perforated bottom of a porcelain funnel is covered with an ordinary, moist, ashless paper filter. The collodion film is placed on the top of this, and pressed down on the plate and slightly around the walls with the help of a suction-pump. By careful adjustment of the film at the corners an air-tight contact is assured. The precipitate is now poured on and filtered in the ordinary way, the filtration being assisted by powerful suction. The collodion film is prepared by pouring some dilute commercial collodion on a polished glass plate, slowly and carefully to avoid the formation of bubbles and to obtain an even layer. When the ether has evaporated, the film may be detached by steeping in water; the prepared films may be preserved for a long time in water. These filters, being extremely thin, free from ash, strong and smooth, fulfil a variety of requirements not satisfied by ordinary paper filters. After drying, the precipitate is easily detached and collected, or it may be incinerated with the film. The films give satisfactory results with the most difficult precipitates, such as colloidal gold, ferric hydroxide, stannic acid, etc., and are useful not only for all ordinary purposes, but also in connection with the Nernst microbalance.

J. F. B.

Comparative Examination of Engler's, Redwood's, and Saybolt's Viscosimeters. W. Meissner. (*Chem. Rev. Fett- u. Harz-Ind.*, 1912, **19**, 30-33, 44-49.)—The efflux period, t_r, measured by Redwood's viscosimeter, and t_s, by Saybolt's universal viscosimeter, may be reduced to Engler's degrees E, by means of the following formulæ:

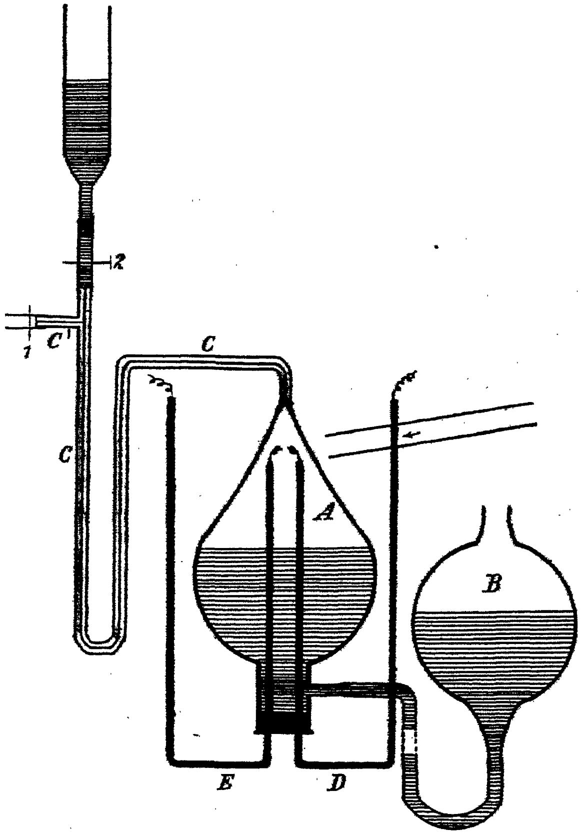
$$t_r = 192{\cdot}2\,k\,(1 + \sqrt{1 + \frac{0{\cdot}01624}{k_2}}$$

$$t_s = 228{\cdot}7\,k\,(1 + \sqrt{1 + \frac{0{\cdot}01309}{k}}.$$

$$k = 0{\cdot}08019\,E - 0{\cdot}07013\,{}^1/E.$$

The values thus calculated agreed within 4 per cent. of those observed with Redwood's apparatus, and within 2 per cent. in the case of Saybolt's apparatus. It is suggested that the results given by Redwood's viscosimeter might with advantage be divided by that given by rape oil, and the quotient regarded as the measure of the viscosity in an analogous fashion to the Engler degrees, for the most carefully purified rape oil must in course of time show alterations in its viscosity. The division of the efflux time by the water value will also increase the accuracy both of Redwood's and Saybolt's apparatus. C. A. M.

Apparatus for Rapid Removal of Nitrogen from Gas Mixtures by Sparking. F. Henrich and W. Eichhorn. (*Zeitsch. angew. Chem.*, 1912, 25, 468-470.)—The best method for the removal of nitrogen from gas mixtures is that of Travers, but, with the apparatus he described, the operation of sparking requires many hours even when quite small volumes of nitrogen have to be removed, and the operation has to be intermittent, as otherwise the glass apparatus is liable to crack in the neighbourhood of the electrodes on account of the high temperature. This latter trouble may be overcome in the apparatus now described by directing a current of air against the pear-shaped bulb in the neighbourhood of the electrodes. But the rapidity with which nitrogen may be removed with the apparatus now described depends more on the means provided for keeping the internal surface of the bulb moistened with caustic soda solution. The apparatus differs from a Hempel pipette only in the arrangement of the electrodes and in the T on the capillary. It is found advantageous to fuse the electrodes through the end of short pieces of special glass (3·5 mm. internal diameter) with a co-efficient of expansion closely approximating that of platinum, and to seal these tubes on to longer tubes of ordinary glass, which are bent in the manner shown,



and filled with mercury. The terminals are of platinum wire 0·35 mm. in diameter, folded on themselves three times as shown. When the apparatus is in continual use, they may be approximated or separated to get any desired length of spark by rotating the glass tubes in the rubber bung. A spark of 0·5 to 1 cm. is employed by the authors. The apparatus is filled with 5 per cent. caustic soda as usual, the clip 2 is closed and gas containing an excess of oxygen admitted at C^1 by opening the clip 1 after connecting to the supply of gas. The induction coil being set working, the clip 2 is opened for a second every few minutes to admit a little caustic soda solution, which spreads as a film over the inner surface of the bulb. For separating small quantities of the rare inert gases from large volumes of nitrogen, it is convenient to have a gas admission tube passing through the rubber bung at the base of the bulb and to admit a mixture of gas and oxygen from time to time, as the liquid in A approaches the level of the terminals. The apparatus will remove 50 c.c. of nitrogen in one hour, and with a more powerful induction coil the speed might be increased. G. C. J.

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REVIEWS.

THE CHEMISTRY OF THE RUBBER INDUSTRY. By HAROLD E. POTTS, M.Sc. Pp. viii + 146. London: Constable and Co., Ltd. 1912. Price 5s. net.

Mr. Potts's volume forms one of a series of "Outlines of Industrial Chemistry," under the editorship of Guy D. Bengough, M.A., M.Sc., and its object, according to the author, is to "exhibit points of contact between chemistry and the industry." The analyst will naturally be mainly interested in the section dealing with analysis, regarding which Mr. Potts states that "the analytical processes of most importance have been critically described and explained." On the whole the author has very fairly accomplished his purpose, although here and there statements are made which may be fairly criticised. Thus, in dealing with the determination of moisture in raw rubber, it is stated that "drying in vacuum over sulphuric acid is accepted as the standard"; but the reviewer is unaware of any "standard" decision in this regard, and, as a matter of fact, the method occupies so much time that it is, as a rule, impracticable in technical work. In describing the estimation of nitrogen the author implies that the "usual" Kjeldahl process consists in the mercury-potassium-sulphide modification; most analysts would probably apply the term "usual" to the Kjeldahl-Gunning process, which, as a matter of fact, works very well on raw rubber. Mr. Potts rightly points out that the importance of a "direct" method of estimating rubber in the crude variety has been much exaggerated, but nevertheless devotes eight pages to its consideration. The recent work of Hinrichsen on the direct estimation of rubber in vulcanised goods by Hübener's method is scarcely in accord with the author's view that "it . . . appears to yield workable results"; but it is only fair to add that Mr. Potts clearly warns the analyst against "direct" methods.

generally. Broadly speaking, this little volume may be recommended to the analyst as affording an excellent bird's-eye view of the methods available for general rubber analysis as seen from a modern standpoint.

P. SCHIDROWITZ.

COCOA AND CHOCOLATE: THEIR CHEMISTRY AND MANUFACTURE. By R. WHYMPER. J. and A. Churchill. London, 1912. Pp. 315. Price 15s. net.

In this work the author's object has been "to provide a standard book of reference dealing with cacao from its growth till manufactured into cocoa and chocolate, and for the first time to trace the chemical composition of cacao from the fresh beans through their various stages of manufacture during fermentation, drying, and roasting." The volume is divided into three parts: (1) History, Botany, and Agriculture of Cacao; (2) Manufacture of Chocolate and Cocoa Powders; (3) Chemistry of Cacao. In the first chapter is gathered together a brief but very interesting historical account of this deservedly popular foodstuff, which began its Western life in the year 1528, when Cortes, returning from Mexico, brought it back to Europe. It was Linnæus, in 1720, who bestowed on it the dignified name of "Theobroma cacao," and the "Food of the Gods" has never gone back in public esteem; for while the consumption of coffee in the United Kingdom has remained stationary from 1899 to 1908, that of cocoa has increased in the same interval from 39,000,000 to 56,000,000 pounds. Not a few people may be surprised to learn that even cocoa, like alcohol, has not escaped scandal; for in 1624 a book was published in Vienna wherein it is stated that consumption of chocolate was largely responsible for many excesses, especially in the case of monks, for whom it was urged its use should be forbidden; and even as late as 1712 the *Spectator* wrote: "I shall also advise my fair readers to be in a particular manner careful how they meddle with romances, chocolate, novels, and the like inflamers, which I look upon as very dangerous to be made use of during this great carnival" (the month of May). Chapters II. to VII. are concerned with what may be called the natural history of cocoa and its preparation for the Western market, and this section contains numerous excellent photographs of picking and curing the beans. A chapter on the chemical composition of cacao beans brings the first section to a close. It is at this point that a protest appears necessary, since here for the first time, and again later, there are printed tables of analyses which look very well until they are compared with other tables by different investigators, each of whom works with his own methods and expresses his results in his own way, such as "starch," "extractives," "fibre or cellulose," etc. Unless some clue is forthcoming, which it seldom is, as to what each particular analyst chooses to label by these names, and, still more important, what his methods were, confusion is bound to result. The author appears to be conscious of this difficulty, for on p. 66 he has this optimistic but scarcely satisfying sentence: "Any variations which may be observed in the figures of analyses made by different individuals may be attributed to the varying quality of the cacaos examined, which, as has been previously pointed out, is largely dependent upon the conditions under which it is grown, and the processes to which the beans have been subjected." And yet eleven pages later (commenting on Table XI.) he says: "It is somewhat difficult to reconcile the results obtained by these two investigators, for the values obtained for albumin, starch, cellulose, and theobromine, are widely different."

Coming to the preparation of cocoa for the table, the author, it is seen, is amongst those who believe that "the nibs of cocoa beans alone should be used for cacao preparations, and that the inclusion of husk must be looked upon as adulteration." For the detection of added shell in cocoa powders, he would appear to attach the greatest importance to the pentosan determination, a complete investigation of which was communicated to the International Congress of Applied Chemistry by R. Adan, 1909 (ANALYST, 1911, **36**, 293).

The detection of "added shell" is of course one of the thorny questions of food analysis, the multiplicity of methods and continual suggestions for fresh ones alone being an indication that all is not quite satisfactory in this direction; and when, after enumerating methods by Filsinger, Weender, Tollens, König, Goske, Adan, and others, the author says that, "whatever the method used, the values obtained are a clear indication of the extent of adulteration of cacao preparations with husk," one can only feel admiration for his clear-sightedness. Concordant results can with difficulty be obtained for "cellulose" unless a more definite method for its estimation be employed than that described on p. 282, where boiling with "dilute mineral acid" and "warming with 1 per cent. soda" are recommended.

The U.S.A. official method for determining alkalinity of ash is described, "the value being expressed as the number of c.c. of $\frac{N}{10}$ sodium hydroxide to neutralise the ash obtained from 1 grm. of cocoa." This should, of course, read $\frac{N}{10}$ acid.

It is to be regretted that the author has not substituted for the tedious process of Dubois the useful polarimetric method of Lintner as modified by Ewers for the estimation of starch, or the space given to Dragendorf's obsolete method might thus advantageously have been used. Fuller details as to the analysis of milk chocolates would have been welcome, since the determination of milk solids and their proportions in chocolate is by no means an easy matter; while help might also have been afforded in dealing with some of the medicated chocolates and cocoas now so often met with.

In addition to the illustrations in the earlier part of the work already alluded to, numerous plates of plant and machinery are provided, and it is unfortunate that the single photomicrograph showing "pure cocoa" is so badly out of focus; in fact, all that can be made out are some large spiral vessels. The author states that "spiral bundles previously referred to as appearing in the shell are also to be found in the kernel," and that "pure cocoa will show isolated spirals," but must not contain "bundles of spirals typical of those appearing in the husk." No means, unless it is their "isolation," are suggested for their respective identification, and, in view of the very general practice of considering the presence of spiral vessels in cocoa as indicative of added shell, it would be interesting to learn the authority for the assertion that they really occur at all in the true kernel. Of course, the absence of these vessels does not in itself prove that no admixture of finely-ground husk has been made, since with modern refinements it is possible so to "fractionate" the husk by wind-sifting and otherwise, that the coarser elements are removed.

The print and general appearance of the volume are excellent; a good index and most useful bibliography of the literature add appreciably to its value; while practically every statement and table is provided with a footnote giving the publication from which it was derived. Recent works, such as Booth, Cribb, and Richards' long paper on chocolate (ANALYST, 1909, 34, 134) and the last addition of Allen, have been consulted, though some of the tables of analyses quoted have little more than an antiquarian interest, such, for example, being those from the Royal Society of Arts for 1874.

H. F. E. HULTON.

EIGHTH
International Congress of Applied Chemistry,
WASHINGTON AND NEW YORK,
September 4th to September 13th, 1912.

British Organising Committee.

Hon. President: THE RIGHT HON. SIR HENRY E. ROSCOE, F.R.S.

President: SIR WM. RAMSAY, K.C.B., F.R.S.

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Hon. Treasurer: DR. M. O. FORSTER, F.R.S.,
84, Cornwall Gardens,
South Kensington, S.W.

The Eighth International Congress of Applied Chemistry will be opened by the President of the United States at Washington, D.C., U.S.A., on Wednesday, September 4th, 1912. The other meetings will be held in New York from Friday, September 6th, to September 13th, 1912. There will be many entertainments during the week, and, afterwards, visits will be paid to factories and other places of interest throughout the United States and in parts of Canada.

As far as possible, all papers presented to the Congress will be printed and distributed before the opening session, and must reach Dr. Bernhard C. Hesse, Secretary, 25, Broad Street, New York City, not later than July 1st next. Rules may be obtained from the Hon. Secretary of the British Organising Committee.

A pamphlet (No. 3), containing general information, programme, details of excursions and visits to works, with forms of application for membership, may be obtained from the Hon. Secretary.

Persons contemplating membership in the Congress can obtain membership tickets from the Treasurer of the Congress, Wm. J. Matheson, 182, Front Street, New York City, by forwarding to him an application for membership and the membership fee, which is $5.00.

Membership tickets can also be obtained from the *Hon. Treasurer of the British Organising Committee*, who will accept £1 as the equivalent of $5.00.

JUNE, 1912. Vol. XXXVII., No. 435.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The monthly meeting of the Society was held on Wednesday evening, May 1, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. H. T. Cranfield, E. G. Jones, M.Sc., F.I.C., and W. H. Roberts, M.Sc., F.I.C., were read for the second time; and certificates in favour of Messrs. Stanley Winter Collins, B.Sc. (Lond.), F.I.C., Assistant Lecturer in Chemistry, King's College, London; and Charles Bluthner Lessner, Analytical and Metallurgical Chemist, Carril, Spain, were read for the first time.

Messrs. R. F. Easton and L. Goodban, A.I.C., were elected members of the Society.

The following papers were read: "The Analysis of Lithopone," by W. L. Austin and Charles A. Keane, Ph.D., F.I.C.; "The Effect of Calcium on the Ammonium Molybdate Lead Assay," by C. O. Bannister and W. McNamara; "The Constituents of Oil of Savin," by J. Watson Agnew, F.I.C., and Robin B. Croad; and "The Detection of Heavy Petroleum in Paints and Vegetable Oils," by W. B. Pollard, B.A.

* * * * *

THE SEPARATION OF ARSENIC FROM ANTIMONY AND OTHER METALS, WITH SOME APPLICATIONS TO TOXICOLOGICAL WORK.*

By STANLEY W. COLLINS, B.Sc., F.I.C.

(*Read at the Meeting, April* 3, 1912.)

Friedheim and Michaelis (*Ber.*, 1895, **28**, 1414) have described a method for quantitatively separating arsenic from vanadium, molybdenum, and tungsten, by distillation with methyl alcohol in a current of hydrogen chloride. Under these conditions it is stated that arsenic trioxide forms a volatile methyl ester. In order to estimate the arsenic the distillate was treated with nitric acid and evaporated to dryness. The residue was dissolved in water, the solution made alkaline with ammonia, and the arsenic precipitated by magnesia mixture and weighed as magnesium pyroarsenate. In estimating arsenic pentoxide no reducing agent need be added, as it is reduced in presence of methyl alcohol and hydrogen chloride, with the formation of the volatile methyl arsenious ester.

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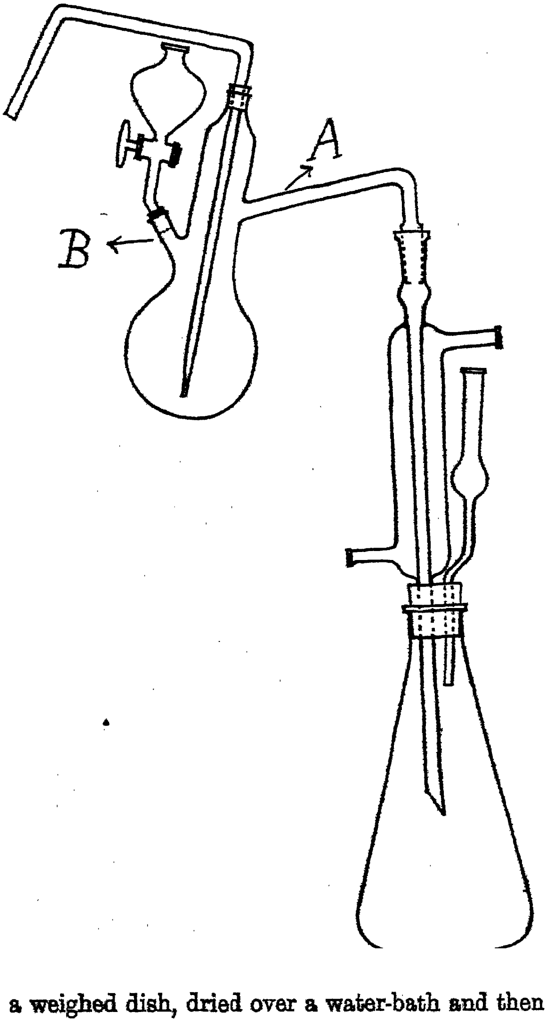
* This work was carried out under the provisions of the Analytical Investigation Scheme.

Cantoni and Chautems (*Annal. de Chim. anal.*, 1905, **10**, 213-214) proposed to separate arsenic from antimony by passing a current of dry air at the ordinary temperature through a solution of arsenic trioxide in hydrochloric acid to which methyl alcohol had been added. They state that under these conditions the arsenic can be completely distilled, leaving the antimony in the residue. It appeared desirable to determine the possible application of these methods to the separation of arsenic from antimony, particularly when these elements are present with organic matter.

The following form of apparatus (see illustration, p. 231) was devised and found to be satisfactory for the experiments involving distillations :

A pear-shaped distilling flask of about 300 c.c. capacity, with a wide neck about 4 cms. diameter and 8 cms. long, drawn down at the top for a small cork or ground joint through which passes the tube for delivery of hydrogen chloride. The flask is provided with a wide side-tube, *A*, about 1 cm. internal diameter, at about 4 cms. from the lower end of the neck. This tube is bent upwards at an angle of about 60 degrees with the neck, so that during distillation it is inclined upwards from the flask, which makes an angle of about 30 degrees with the vertical. This arrangement allows of rapid distillation, whilst it prevents the solution being carried over by spurting. The inlet tube *B*, of about 1 cm. diameter and 2 cms. length at the lower end of the neck, serves for the introduction of substances to be analysed, and into it, before the distillation is commenced, a tap-funnel is fitted by means of a cork or a ground joint, to allow of the addition of more methyl alcohol. In a later form of apparatus the inlet tube *B* was enlarged to about 1½ cms. diameter, thus making it more convenient for the introduction of substances to be analysed. The tap funnel was also ground in. The side-tube was ground into a Liebig's condenser, and bent so that, during an experiment, the condenser is in a position slightly sloping away from the distilling flask. The inside tube of the condenser is about 1 cm. in diameter and 40 cms. in length, passing through a cork into a 30-ounce conical flask. Through another hole in the cork a tube filled with glass beads is fitted. The jacket of the condenser is about 17 cms. long.

Several experiments were carried out on the lines proposed by Cantoni and Chautems (*loc. cit.*), but the method proved to be of no value ; for under the conditions described by these chemists only a small proportion of arsenic trioxide could be distilled, even when a current of dry air was passed for four hours. A more successful experiment was carried out by the method of Friedheim and Michaelis (*loc. cit.*). 0·2 grm. of arsenic trioxide was washed into the distillation flask with 50 c.c. of methyl alcohol, which was then saturated with dry hydrogen chloride. The flask was then heated on a water-bath ; the distillation was repeated twice with 50 c.c. of methyl alcohol, a slow stream of hydrogen chloride being passed during the distillation. On diluting the residue in the flask and passing in sulphuretted hydrogen, no arsenious sulphide was precipitated. The method used in this and many subsequent experiments for the estimation of arsenic in the distillate, consisted of saturating with sulphuretted hydrogen, after diluting with an equal volume of water, washing the precipitated arsenious sulphide with hot water, alcohol, a mixture of carbon disulphide and alcohol, alcohol, and then hot water. The precipitate was then washed from the filter-paper into a weighed dish, dried over a water-bath and then at 105° to 110° C., and weighed.



The precipitate was tested for antimony by digesting with strong hydrochloric acid, filtering, diluting and saturating with sulphuretted hydrogen. The antimony sulphide was dissolved in hydrochloric acid with a little potassium chlorate, and antimony obtained from the solution by means of pure iron wire.

Arsenic in the distillate was also estimated satisfactorily by titration with standard iodine solution. An experiment was made on 0·2 grm. of arsenic trioxide, with 0·2 grm. of antimony trioxide, with the result that about 0·02 grm. of antimony trioxide was found with the distilled arsenious oxide. It will thus be seen that the formation of antimony chloride and its subsequent volatility render the method inapplicable to the quantitative separation of arsenic from antimony. Many experiments were made in order to overcome this difficulty, in the course of which it was found that the volatility of arsenic trioxide in the vapour of methyl alcohol was considerably increased by the addition of strong sulphuric acid or phosphorus pentoxide to the arsenic trioxide and methyl alcohol before distillation; neither arsenic pentoxide nor antimony trioxide could be distilled under these conditions. Attempts were made to devise a method based on these facts for the quantitative separation of arsenic from antimony, but it was found that the removal of the last traces of arsenic was a tedious process, in which it was necessary to use a comparatively large volume of methyl alcohol. A complete separation was, however, effected by taking a mixture of methyl alcohol and the oxides and a sufficient quantity of sulphuric acid, and passing a stream of hydrogen chloride through for some time before commencing the heating. The passage of the gas was continued throughout the distillation. Under these conditions it was found that the arsenic was completely distilled, whilst the more basic antimony was retained by the sulphuric acid. When phosphorus pentoxide was substituted for sulphuric acid in these experiments, small quantities of antimony were carried over with the arsenic.

The following account of typical experiments illustrates the application of this method:

0·08 grm. of arsenic trioxide and 0·08 grm. of antimony trioxide were washed into the distilling flask with 30 c.c. of methyl alcohol and a mixture of 6 c.c. of sulphuric acid (sp. gr. 1·84) with 20 c.c. of methyl alcohol. Hydrogen chloride was passed in for ten minutes and the flask heated on a water-bath, the distillate being collected in a 30-ounce conical flask into which a small quantity of water was occasionally poured over the glass beads. The distillation was repeated twice with 50 c.c. of methyl alcohol. The arsenic in the distillate was precipitated by sulphuretted hydrogen, washed, and weighed; 0·0796 grm. was found. No antimony sulphide could be detected in the precipitate. This analysis was repeated, using 0·2 grm. of arsenious oxide and 0·2 grm. of antimony oxide with 8 c.c. of sulphuric acid (sp. gr. 1·84). 0·200 grm. of arsenic trioxide was found. In the next experiment the conditions were the same, with the exception that the arsenic in the distillate was titrated with iodine, after adding an excess of sodium bicarbonate. 0·200 grm. of arsenic trioxide was found.

2·0 grms. of antimony oxide with 0·1 grm. of arsenic trioxide were mixed with 15 c.c. of sulphuric acid (sp. gr. 1·84) and 50 c.c. of methyl alcohol. The distillation was repeated with the following volumes of methyl alcohol, 50 c.c., 50 c.c., 20 c.c., 20 c.c., when, on testing the distillate with sulphuretted hydrogen, no precipitate was obtained. 0·1002 grm. of arsenic trioxide was found. The precipitate was free from antimony.

3·0 grms. of antimony oxide with 0·1 grm. of arsenic trioxide were mixed with 15 c.c. of sulphuric acid (sp. gr. 1·84) and 50 c.c. methyl alcohol. Distillation was repeated as in the last experiment. 0·101 grm. of arsenic trioxide was found. In this case the precipitate contained antimony. The experiment was repeated, using 3·0 grms. of antimony oxide with 0·06 grm. of arsenic trioxide and a smaller quantity of sulphuric acid—*i.e.*, 10 c.c. A precipitate equivalent to 0·064 grm. of arsenic trioxide was found. Antimony was present in the precipitate.

The application of this method to the separation of arsenic pentoxide from antimony was next investigated. It was found that the arsenic from arsenic pentoxide could be distilled completely only in the presence of a reducing agent such as ferrous chloride. The following experiments show the results obtained for the estimation of arsenic pentoxide; the first one gave a satisfactory result for the separation of arsenic from other metals of the sulphuretted hydrogen group as well as from antimony:

0·06 grm. of arsenic pentoxide with 0·05 grm. of antimony oxide, 0·04 grm. of bismuth oxide, 0·03 grm. of mercurous chloride, 0·03 grm. of mercuric chloride, and 5 grms. of ferrous chloride, were introduced into the distilling flask with 50 c.c. of methyl alcohol. To these were added 10 c.c. of sulphuric acid (sp. gr. 1·84); hydrogen chloride was passed through the contents of the flask before and during the distillation, which was repeated twice with 50 c.c. and twice with 20 c.c. of methyl alcohol. The distillate was saturated with sulphuretted hydrogen and the precipitated arsenious sulphide, washed, dried, and weighed, as before. The weight of arsenic pentoxide found was 0·0596 grm. The precipitated arsenious sulphide was found to be free from antimony, bismuth, and mercury.

0·1 grm. of arsenic pentoxide with 2 grms. of antimony oxide was mixed in the distillation flask with 15 c.c. of sulphuric acid (sp. gr. 1·84), 50 c.c. of methyl alcohol, and 5 grms. of ferrous chloride. Hydrogen chloride was passed before and during distillation, which was repeated twice with 50 c.c. and twice with 20 c.c. of methyl alcohol. The weight of arsenic pentoxide found was 0·0998 grm., and there was no antimony in the precipitate.

These results indicate that this method is applicable, under suitable conditions, to the quantitative separation of arsenic from antimony and from other metals. Before pointing out the conditions necessary for this method, it is essential to give an account of an experiment which shows the influence of water on the volatilisation of antimony trichloride: 1 grm. of antimony oxide was mixed with 10 c.c. of sulphuric acid (sp. gr. 1·84) and 50 c.c. of water. Hydrogen chloride was passed in for about fifteen minutes as well as during the distillation. Sulphuretted hydrogen was passed into the distillate, which yielded an appreciable precipitate of antimony sulphide. To about 20 c.c. of the residual solution an equal volume of methyl alcohol was added, and hydrogen chloride passed through as before. Separate small volumes of the distillate were collected and saturated with sulphuretted hydrogen. A barely perceptible coloration was obtained in the first portions, but, as the alcohol was distilled off, the quantity of antimony sulphide precipitated became appreciable. It is evident, therefore, that the material used in this method should be sufficiently free from water, since, in presence of water, sulphuric acid fails to retain the antimony completely. With regard to the amount of sulphuric acid necessary to retain the antimony, 10 c.c. of acid (sp. gr. 1·84) is a convenient quantity for not more than 1 grm. of antimony oxide; whilst 15 c.c. of acid will retain up to 2 grms. of antimony oxide, and is the maximum quantity convenient to use with 50 c.c. of methyl alcohol, since with more than this amount the distillation becomes too slow. An experiment was next made in order to gain some idea of the maximum amount of arsenic trioxide which could be volatilised, using 50 c.c. of methyl alcohol. Arsenic trioxide 5 grms., with 10 c.c. of sulphuric acid, and 50 c.c. of methyl alcohol, were distilled in a stream of hydrogen chloride in the usual manner, and 0·96 grm. of arsenic trioxide was found to have been volatilised, an amount largely in excess of that used in any estimation. In the quantitative separation of arsenic from antimony, when not more than 0·2 grm. of arsenic trioxide was used, about 60 per cent. of the arsenic was volatilised in the first 50 c.c. of alcohol; a further 50 c.c. generally carried over all but a small amount, which was volatilised in the last 50 c.c. In presence of a large excess of antimony, it is advisable to add a further 10 c.c. of methyl alcohol and to test the distillate with sulphuretted hydrogen, as an additional 10 or 20 c.c. of methyl alcohol may be necessary to remove the last traces of arsenic.

Pure methyl alcohol, not specially dehydrated, was used in all these experiments. Commercial methyl alcohol is unsatisfactory, as the acids employed induce the formation of compounds of high boiling-point which retard the volatilisation of the arsenic. In an experiment in which ethyl alcohol was used, instead of methyl alcohol, the quantity of arsenic carried over was about half as great as would have been volatilised by the same volume of methyl alcohol.

It has been stated in this paper that the quantity of arsenic trioxide which could be distilled over was increased by mixing sulphuric acid with the alcohol and the oxide in the distillation flask, and that neither arsenic pentoxide nor antimony trioxide could be distilled in this manner. This suggests an easy test for arsenic trioxide in the presence of antimony as well as other metals. This method could only be applied as a test for arsenic trioxide in the presence of arsenic pentoxide when certain organic and other reducing agents were excluded.

The following experiments were made to test this method. Equal weights of the following substances were mixed—mercurous chloride, mercuric oxide, stannous chloride, cadmium oxide, antimony oxide. About 1 grm. of this mixture was placed in a small distilling flask, and 20 c.c. of methyl alcohol and 3 c.c. of sulphuric acid were added. The flask was attached to a condenser and heated by a flame, the distillate being collected in a small flask containing about 5 c.c. of water. On passing sulphuretted hydrogen through the distillate no precipitate was obtained. This experiment was repeated, using 1 grm. of the mixed substances with 0·3 grm. of arsenic pentoxide. No precipitate was obtained on passing sulphuretted hydrogen through the distillate. The experiment was again repeated, using 1 grm. of the mixed substances with 2 mgrms. of arsenic trioxide. A precipitate of arsenious sulphide was obtained on passing sulphuretted hydrogen through the distillate.

Application of this Method to the Separation of Arsenic from Antimony and Other Metals in the Presence of Organic Matter.

The character of the material for analysis will determine the mode of application. The separation can be effected without previous destruction of the organic matter if the arsenic be not intimately combined with the organic matter. The material should also be fairly dry.

The following experiment is an example of these conditions: 20 grms. of powdered biscuit material were placed in the distillation flask; 0·1 grm. of arsenic trioxide and 0·1 grm. of antimony oxide were washed into the flask with a mixture of 50 c.c. of methyl alcohol and 8 c.c. of sulphuric acid. Hydrogen chloride was then passed through the contents of the flask for about ten minutes. The flask was heated on the water-bath, a stream of hydrogen chloride being continued during the distillation, which was repeated twice with 50 c.c. and twice with 20 c.c. of methyl alcohol. The arsenic was precipitated from the distillate by means of sulphuretted hydrogen, washed, dried, and weighed, and an amount equivalent to 0·0986 grm. of arsenic trioxide was found.

For the next experiments I obtained specimens of viscera from a case which had been treated with salvarsan. In the first test 50 grms. of the kidney were heated in a Jena distillation flask with sulphuric acid, and the water distilled off as completely as possible. Then methyl alcohol was added and hydrogen chloride passed through the viscid contents of the flask, and the distillation effected as before. This method was not satisfactory. Ninety-eight grms. of the liver were treated with 10 c.c. of nitric acid (sp. gr. 1·4), and charred in the usual manner with sulphuric acid. The charred mass was washed into the distilling flask with 50 c.c. of methyl alcohol, and about 5 grms. of ferrous chloride were added. Hydrogen chloride was then passed in as usual, and the flask heated on the water-bath. The distillation was repeated twice with 50 c.c. and twice with 20 c.c. of methyl alcohol. The distillate was saturated with sulphuretted hydrogen, and the precipitated arsenious sulphide was purified by dissolving in ammonia and reprecipitating. The arsenious sulphide was then dissolved in nitric acid, the solution made alkaline with ammonia, and the arsenic precipitated as magnesium ammonium arsenate, and finally weighed as the pyroarsenate. Weight of arsenic trioxide per 100 grms. of the liver: From the sulphide, 3·6 mgrms.; from the pyroarsenate, 3·1 mgrms. The residue was found to be free from arsenic.

I am indebted to Mr. John Webster for the result of an estimation of arsenic in another portion of this liver. This estimation was carried out, in Dr. Willcox's laboratory, by the usual extraction method, the arsenic being finally weighed as magnesium pyroarsenate. Weight of arsenic trioxide found per 100 grms. of viscera was 3·3 mgrms. This result was confirmed by an estimation made by the Marsh-Berzelius method.

Attempts to confirm the statement of Friedheim and Michaelis (*loc. cit.*), that the volatility of arsenic trioxide in methyl alcohol and hydrogen chloride is due to the formation of a methyl arsenious ester, were unsuccessful. An excess of arsenic trioxide was heated with dehydrated methyl alcohol and sulphuric acid for some hours, also methyl alcohol with arsenic trioxide was treated with hydrogen chloride, and also with hydrogen chloride and sulphuric acid. In each experiment the distillate was fractionally redistilled, with the result that the boiling-point did not rise appreciably above that of methyl alcohol. The compounds of arsenic with alcohol radicles have been investigated by the following: J. M. Crafts (*Bull. Soc. Chim.*, 1870, **14**, 99) obtained arsenates and arsenites of the alkyl radicles of the general formulæ R_3AsO_4 and R_3AsO_3. For the arsenites he used three methods involving the interaction in a sealed tube of (1) ethyl iodide and silver arsenite at 150° C.; (2) arsenic bromide and sodium ethoxide; (3) arsenic trioxide and ethyl silicate at 200° C. The boiling-point of methyl arsenite is 128° C. Auger (*Compt. rend.*, 1902, **134**, 238, and 1906, **143**, 907) states that he obtained small yields of esters by heating arsenic trioxide and alcohol together in a sealed tube for some hours, although he did not succeed in isolating the esters. The maximum amount of esterification in the case of methyl alcohol is given as 6·5 per cent. The method of estimation of the amount of esterification appears not to be very satisfactory (*cf. J. Chem. Soc.*, 1908, **93**, 1367). In experimenting with propyl, normal butyl, and isobutyl alcohol with arsenic trioxide, he states that good results were obtained by removing the water formed either by fractional distillation or by passing the alcohol and water vapour over calcium carbide in an adapter, when the water is removed and the alcohol returns to the flask in which the reaction is taking place.

W. R. Lang, J. T. Mackey, and R. A. Gortner (*J. Chem. Soc.*, 1908, **93**, 1364) obtained a yield of 33·8 per cent. of methyl arsenite by heating methyl alcohol and arsenic trioxide together in the presence of anhydrous copper sulphate under a reflex condenser, and fractionating the resulting liquid under diminished pressure. They state that they have prepared methyl arsenite by a similar method without the anhydrous copper sulphate. No statement is made as to yield obtained.

J. T. Mackey (*J. Chem. Soc.*, 1909, **95**, 605) prepared esters of the aliphatic alcohols with antimony trioxide, using anhydrous copper sulphate as a dehydrating agent. The boiling-point of methyl antimonite is given as 65° C.

Some esterification appears therefore to take place when arsenic trioxide and methyl alcohol are heated together, the amount of ester formed being increased in the presence of a desiccating substance such as anhydrous copper sulphate. However, under the conditions which prevailed during these estimations no evidence has been obtained for the formation of an arsenious ester. I propose to further examine the possibility of ester formation involved in the separation described in this communication.

In conclusion, I desire to express my thanks to Dr. W. H. Willcox and Mr. John Webster for their practical interest and help, and to Mr. A. Chaston Chapman for the help he has afforded me throughout the course of this work.

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DISCUSSION.

The PRESIDENT, having invited discussion,

Mr. CHASTON CHAPMAN suggested that the function of the sulphuric acid might be not only to retain the more basic antimony, but also to assist esterification. He did not quite know upon what evidence Mr. Collins based his opinion that no formation of ester occurred, and he (Mr. Chapman) could not help feeling that the separation was in reality dependent on the formation of a methyl arsenious ester. With the comparatively small quantities of arsenic used in Mr. Collins' experiments, the detection in the distillate of such an ester would not be easy; but it was quite clear that its formation would be favourably influenced by the presence of the sulphuric acid.

Mr. C. REVIS said that this process seemed to be a great advance on that of Clark. He had had occasion to use Clark's process a good deal some years ago, but had not been able to obtain very satisfactory results with it, finding that antimony was carried over during the distillation, especially when the antimony was large in quantity as compared with the arsenic. When, however, no other metal but arsenic was present, the results were fairly good.

Dr. WILLCOX said the process was itself an old one, and he (Dr. Willcox) believed that Taylor had claimed to be the originator of a modification of it for toxicological purposes. The process in that form, however, involved considerable risk of explosion, and it was not reliable for the separation of arsenic from small quantities of antimony and tin. Mr. Collins had tested his form of the process very thoroughly, and he (Dr. Willcox) believed that, if carried out under the conditions which Mr. Collins laid down, it would be found to be reliable for the separation of arsenic from antimony, mercury, bismuth, etc. It would be very useful for toxicological work, especially in criminal cases of arsenical poisoning, because in such cases doses of bismuth were frequently given, and there was considerable difficulty in the analysis of viscera containing arsenic accompanied by large quantities of bismuth when the usual method of precipitation as sulphides was employed. Apart from the question of the separation of arsenic from antimony, etc., this process would be valuable in dealing with arsenic alone, as was shown by the results that had been obtained in the case of salvarsan-poisoning referred to.

Mr. T. MACARA said that the process devised by the late Dr. Clark was intended for use in connection with white metals, bronzes, and similar materials, and for such purposes it gave very good results when properly applied. It was essential to use strong hydrochloric acid, and not to concentrate the solution too much and so unduly raise its boiling-point. The one fault of the process was that it necessitated two or three distillations, but that difficulty might be overcome by passing hydrochloric acid gas into the distilling flask, as suggested by Mr. Collins, which would enable good results to be obtained at a comparatively low temperature.

The PRESIDENT said that it seemed to him that Mr. Collins' process was essentially different from that of Dr. Clark. Of course, in a toxicological investigation it would not be proper to assume that a volatile product was arsenic without weighing it as a definite arsenical compound; but for many purposes it would probably be sufficient to boil the sulphide of arsenic with water, and titrate the arsenious oxide with iodine.

Mr. COLLINS, in reply, said that the statement of Frankheim and Michaelis was that the method depended on the formation of a methyl arsenious ester, but so far as his own work had gone he had not been able to obtain any evidence of the formation of such an ester. He had, however, only as yet been able to make one experiment as to this, which showed that the boiling-point of the distillate was not higher than that of methyl alcohol, whereas the boiling-point of pure methyl arsenite was 128° C. In this experiment dehydrated methyl alcohol and sulphuric acid were heated with arsenic trioxide, and hydrochloric acid gas was passed through; on fractional distillation it was found that the boiling-point did not rise appreciably above that of methyl alcohol. He hoped later on to make further experiments by carrying out the fractional distillation under reduced pressure, under which conditions some esterification might possibly take place.

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THE ANALYSIS OF LITHOPONE.*

BY W. L. AUSTIN AND CHARLES A. KEANE, D.Sc., PH.D., F.I.C.

(*Read at the Meeting, May* 1, 1912.)

THE chief estimations required for the technical valuation of lithopone comprise the determination of the zinc sulphide, zinc oxide, and barium sulphate. Several other zinc compounds have been shown to be present, in varying amounts, in the mixture, such as the carbonate, normal sulphate, and basic sulphate. Coffignier (*Bull. Soc. Chim.*, 1902, **27**, 829, 943) has also drawn attention to the occurrence of soluble salts of zinc and of the hydroxy-sulphide $Zn\langle\begin{smallmatrix}OH\\SH\end{smallmatrix}$ in cases where the product has been imperfectly calcined; but such constituents are to be regarded as abnormal, and should not be present in lithopones that have been properly prepared and washed. In addition, lithopone sometimes contains a small proportion of barium carbonate, and traces of impurities, including ferric oxide, alumina, lime, and magnesia. Whilst the determination of these secondary products is seldom required, their presence is of importance in selecting a method of analysis. This is especially the case in regard to the influence of the iron and calcium on the estimation of the zinc, and also in regard to the possible presence of zinc compounds, other than the sulphide and oxide, as any soluble salts of zinc are, of course, of no value as a pigment. The presence of barium carbonate can be readily determined by a separate estimation if required.

The numerous methods that have been proposed for the analysis of lithopone consist in the determination of the total zinc, the total sulphide, and the barium sulphate. By calculating the total sulphide to zinc sulphide, and subtracting its equivalent in zinc from the total zinc found, the proportion of zinc other than sulphide is calculated and generally expressed as oxide. As a check on this last factor, the zinc oxide and carbonate can be extracted either with dilute acetic acid or with a mixture of sodium acetate and acetic acid, and estimated separately.

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* This work was carried out under the provisions of the Analytical Investigation Scheme.

These determinations have involved simply a combination of ordinary methods of analysis, of which the following may serve as examples:

P. Drawe (*Zeitsch. angew. Chem.*, 1902, **15**, 174, 229, 297) estimates the total zinc by precipitation as carbonate and the zinc present as sulphide by treating the sample with 1 per cent. acetic acid, to remove the oxide and carbonate, and precipitating the remaining zinc again as carbonate. Kochs and Seyfert (*Zeitsch. angew. Chem.*, 1902, **15**, 802) precipitate the total zinc as sulphide, and estimate the oxide and carbonate by solution in 5 per cent. acetic acid and precipitation as sulphide. Amsel (*Zeitsch. angew. Chem.*, 1902, **15**, 174) also extracts the oxide with 5 per cent. acetic acid, and estimates both it and the total zinc volumetrically by titration with calcium ferrocyanide. For the estimation of the sulphide-sulphur as an indirect means of determining the content of zinc sulphide, J. F. Sacher (*Farbenzeitung*, 1910, **15**, 1810) recommends treatment with metallic tin and hydrochloric acid, passing the evolved sulphuretted hydrogen into an ammoniacal solution of hydrogen peroxide, and precipitating the ammonium sulphate formed with barium chloride, whilst H. Wolff (*Farbenzeitung*, 1910, **15**, 1859) estimates the sulphuretted hydrogen volumetrically by absorbing it in iodine solution, and titrating back the excess of the latter with thiosulphate. J. S. Remington and C. Smith (*Caoutchouc and Gutta-Percha*, 1909, **6**, 3259) estimate the sulphide-sulphur by oxidation with bromine and precipitation as sulphate, and the total zinc as carbonate, or, if salts of iron, calcium, or barium (soluble in hydrochloric acid) are present, as sulphide.

The alternative to these many proposals that we recommend is to estimate the total zinc volumetrically and the zinc sulphide indirectly by oxidising the sulphide-sulphur to sulphate. The gravimetric estimation of the zinc as carbonate is inaccurate in presence of calcium and iron, both of which are usually present, and precipitation as sulphide is a tedious determination. A direct estimation of the sulphide-sulphur is especially desirable, as the value of the pigment is chiefly dependent on the proportion of zinc sulphide present, and of the various methods available oxidation followed by precipitation is simpler and easier to carry out than the determination of the sulphuretted hydrogen evolved on decomposition.

For the volumetric estimation of the zinc we have employed a method devised by A. Voigt (*Zeitsch. angew. Chem.*, 1889, **2**, 307) for the analysis of zinc ores and products which is sufficiently accurate for the estimation, and which has the advantage that the results are not affected by the presence of iron or calcium. For the oxidation of the sulphide-sulphur either bromine or nitric acid may be used; we have found the former preferable, and carry out the estimation on the same lines as proposed by Remington and Smith (*loc. cit.*). Whilst, therefore, there is no claim to originality in the methods employed, we have found the combination adopted more rapid and adaptable than previous proposals, and sufficiently accurate for technical purposes.

The method of analysis is as follows: 5 grms. of the finely-ground sample are treated with concentrated hydrochloric acid and a little potassium chlorate, the whole evaporated to a small bulk on the water-bath, taken up with boiling water, and the residual barium sulphate filtered off and thoroughly washed. The filtrate is collected in a 250 c.c. graduated flask, made up to the mark, and aliquot portions taken for the estimation of the total zinc by Voigt's method. The potassium ferrocyanide solution is made up by dissolving 46 grms. of the crystallised salt in a litre of water, and is standardised by a solution of zinc chloride containing 12·4476 grms. of pure zinc oxide per litre; 1 c.c. of this solution is equivalent to 0·01 grm. of zinc. For the estimation, 50 c.c. of the above filtrate are taken, 10 c.c. of a solution of tartaric acid (200 grms. per litre) and 10 c.c. of a solution of ferric chloride (60 grms. per litre) added, the whole diluted to about 200 c.c., made just alkaline with ammonia, and titrated; the completion of the reaction is determined by adding a drop of the solution to dilute acetic acid (1:3) on a pitted porcelain plate until a permanent blue colour is produced. After a preliminary estimation a sharp end-reaction is easily secured, and it is not affected by the presence of an excess of ammonia. In a series of tests made, in which the quantity of ammonia added was increased up to twice the amount necessary for neutralisation, the maximum difference in the titrations did not exceed 0·15 c.c.

For the estimation of the sulphide-sulphur, 0·5 grm. of the sample is oxidised with bromine, after the addition of water, hydrochloric acid added, the residual barium sulphate filtered off, and the sulphuric acid in the filtrate precipitated by barium chloride. By calculating the sulphur thus found to zinc sulphide, and subtracting its equivalent of zinc from the total zinc estimated volumetrically, the content of zinc, other than sulphide, is obtained by difference, as stated above.

The following results were obtained by this method with three different samples of lithopone; the zinc, other than that present as sulphide, is calculated as oxide:

	I.	II.	III.
$BaSO_4$	64·36	67·59	72·80
ZnS	30·98	27·82	24·14
ZnO	0·55	1·82	0·88
CaO, Fe_2O_3, $BaCO_3$, Moisture, etc.	4·11	2·77	2·18
	100·00	100·00	100·00

As a check upon the volumetric estimation of the zinc, that in Sample No. 1 was also estimated by precipitation as sulphide, the result obtained being 21·44 per cent. zinc as against 21·20 per cent. by titration. The indirect determination of the zinc oxide was also checked by treating 2 grms. of each sample with 5 per cent. acetic acid at the ordinary temperature for one hour, with occasional agitation, the dissolved zinc filtered off, precipitated as sulphide, and finally estimated as oxide in the usual manner. The results obtained were as follows:

	I.	II.	III.
ZnO (calculated)	0·55	1·82	0·88
ZnO (estimated by precipitation) ...	0·75	1·70	0·90

These data confirm the reliability of the indirect method of estimation, and also show that the zinc, other than sulphide, in the samples analysed is practically all present as oxide.

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THE SIR JOHN CASS TECHNICAL INSTITUTE, ALDGATE.

Discussion.

The President said that the specimens of lithopone that passed through his hands were usually ground in oil. Those who were experienced in work of this kind would know that it was never possible to extract the oil completely; some oxidised oil always remained unextracted, and this caused difficulty in the estimation of the sulphide sulphur by oxidation with bromine in an acid solution. His practice was first of all to add some caustic soda to the extracted lithopone, and then pour in the liquid bromine and acidify. Under those conditions no globules of sulphur or sulphur bromide escaped oxidation. With regard to the estimation of the zinc, he could understand that, in laboratories where such estimations were made very frequently, it would be worth while to use a volumetric process; but, when it was only necessary to make such estimations occasionally, a gravimetric process was, perhaps, equally expeditious. He should like to ask whether the authors had tried precipitating the zinc in a solution containing a minute trace of free mineral acid. Under those conditions the zinc was completely precipitated, and in a granular form. Of course, during the precipitation more acid was set free, and one must be careful to neutralise all but the very faintest trace. In the analyses given by the authors the proportions of what might be called impurities were larger than he had been accustomed to find.

Mr. T. Macara asked whether the authors had tried the phosphate method of estimating zinc in the case of such a compound as lithopone. He thought that this method, which was proposed by the late Dr. Clark for the estimation of zinc in alloys, etc., would probably work well in such a case as this.

Dr. H. P. Stevens asked whether the lime shown as CaO in the authors' analyses referred to caustic lime. Lithopone was used in rubber mixings, in which the presence of caustic lime was very undesirable.

The President added that a certain amount of soluble SO_3 was always present, and it was his practice to determine this, calculating it as calcium sulphate; and perhaps if that were done in these cases, some, at any rate, of the lime would appear as calcium sulphate. In many cases the quantities of CaO and of soluble SO_3 found were just equivalent to one another, showing that all the lime was present as calcium sulphate.

Mr. J. H. B. Jenkins asked whether the oxide of iron referred to in the analyses amounted to anything appreciable. One of the advantages of lithopone paint was its dead whiteness, and one would not expect this if any sensible quantity of iron were present. The majority of the samples of lithopone that he had examined contained more barium sulphate relatively to the sulphide of zinc than was shown in the authors' analyses. He believed that in the trade the proportions were supposed to be about 3 parts of barium sulphate to 1 part of zinc sulphide.

Dr. Dyer incidentally observed—although, of course, the remark did not apply to genuine "lithopone"—that mixed pigments containing barium sulphate in the form of barytes sometimes contained considerable quantities of calcium fluoride, owing to admixture of fluor spar with the heavy spar from which the barytes was ground. He mentioned this because he had seen no published reference to it, and it was at first puzzling to find a considerable quantity of calcium present in a form other than the familiar carbonate or sulphate.

Dr. Keane, in reply, agreed that the method of estimating zinc as phosphate was an excellent one, but they wished to employ a volumetric rather than a gravimetric method, in order that the process might be as useful as possible for works purposes. He also agreed with the President as to the advantages of precipitating the zinc in the presence of a trace of mineral acid. Up to the present they had not had occasion to deal with small quantities of organic matter, such as would be derived from oil, but, in another connection, he had found that organic matter could be effectively got rid of by electrolysing the solution in presence of dilute nitric acid. In that way, even in the case of animal products containing a good deal of organic matter, a crystal-clear solution could be obtained containing no trace of organic matter. With regard to the lime, this undoubtedly was not present as such, but probably to some extent as sulphate, and as sulphide and carbonate. Of course, by estimating the soluble constituents one could always, if it were desired to do so, calculate the lime to calcium sulphate, as suggested by the President. The quantity of iron present was small, usually no more than a trace. The moisture was occasionally rather high, sometimes as much as 1 per cent. Of the samples analysed, the first was made in Germany, the other two being English products; there are, of course, considerable variations in the relative proportions of barium sulphate and zinc sulphide in lithopones.

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ON THE EFFECT OF LIME ON THE AMMONIUM MOLYBDATE METHOD OF LEAD ASSAY.*

By C. O. BANNISTER, A.R.S.M., and W. McNAMARA.

(*Read at the Meeting*, *May* 1, 1912.)

During the analytical portion of the experimental work in connection with a recent research on the "Theory of Blast Roasting of Galena" (*Bull. Inst. Min. and Met.*, 1912, No. 89), the necessity arose for ascertaining the effect of lime on the ammonium molybdate method of lead assay. On referring to published results on this point, a great divergence of opinion was found to exist, some writers stating that the presence of lime had no influence, and others that the results obtained for lead in the presence of lime were too high.

I. C. Bull (*School of Mines Quart.*, 1902, **23**, 348), who examined the effect of calcium, barium, strontium, antimony, and bismuth on the assay, came to the conclusion that calcium, bismuth, and antimony had no effect on the result, and that barium and strontium caused low results. In this work, however, Bull added the impurities to the solution of lead sulphate in a form in which they could not ordinarily exist in the course of assays, for the calcium, barium, strontium, and antimony were added as chlorides and the bismuth as nitrate.

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* This work was carried out under the provisions of the Analytical Investigation Scheme.

On following out the method as originally devised by H. H. Alexander (*Eng. and Min. J.*, 1893, 55, 298), it is obvious that only insoluble sulphates can be left with the sulphate of lead, and also that the impurities most likely to interfere are calcium, barium, and strontium.

H. A. Guess (*Amer. Inst. Min. Eng.*, 1904, 35, 359) points out that the molybdate method cannot be used with safety in dealing with poor lead ores containing large quantities of lime. W. H. Seamon ("A Manual for Assayers and Chemists," Chapman and Hall, 1910, p. 91) also states that the molybdate method is not reliable on low-grade ores containing much lime, and that it frequently fails when there is iron in the ores, all the sulphate of iron failing to go into solution, and obscuring the end-point with tannin. To overcome these difficulties, Seamon separates the lead by precipitation on aluminium. A. H. Low ("Technical Methods of Ore Analysis," Chapman and Hall, fifth edition, p. 152) states that Alexander's original method did not sufficiently provide for the presence of calcium, which is a frequent constituent of lead ores. Calcium forms a molybdate which is more or less insoluble under the conditions of titration, and tends to raise the results in a rather irregular manner. To overcome this, Low recommends a longer method, involving the separation of the lead from the ammonium acetate solution by means of ammonium sulphide.

In a recent paper on "Quick Combination Methods in Smelter Assays" (*Bull. Inst. Min. and Met.*, 1912, No. 89), A. F. French gives results showing that lime does not interfere with the molybdate assay as ordinarily carried out, and these results are confirmed by F. C. Robinson (*ibid.*, 1912, No. 90), who, in a written contribution to the discussion of French's paper, states that when lime is known to be present the assay, after evaporation to the point at which SO_3 fumes are liberated, is not diluted, cooled, and allowed to stand overnight, but is diluted only when it can be proceeded with at once after cooling, as calcium sulphate crystallises out and is difficult to redissolve. By washing by decantation twice with 30 c.c. of cold dilute sulphuric acid (1 per cent. by volume) and once with water, calcium sulphate is sufficiently well removed.

From the foregoing it is evident that the effect of lime on the assay is by no means clearly understood, and it is also evident that in order to correctly interpret the action of lime on the titration a solution of calcium sulphate in ammonium acetate should be used.

Preliminary experiments showed that a solution of calcium sulphate in ammonium acetate does not form an insoluble molybdate with ammonium molybdate in the absence of lead, neither does it react with the lead molybdate formed during an ordinary lead assay; for if the assay be carried out to a satisfactory end-point, and then a quantity of solution of calcium sulphate in ammonium acetate be added, the end-point is found to be permanent, no more ammonium molybdate being required to react with the lime. If, however, a solution containing sulphates of lead and lime in ammonium acetate be titrated with ammonium molybdate solution, then the lime causes a larger amount to be required, thus giving high results, the excess necessary increasing with the lime added to a certain point, after which additional lime makes no difference.

The following tables indicate the effect of the addition of increasing amounts of calcium sulphate:

TABLE I.—*Effect of Small Quantities of Calcium Sulphate on the Ammonium Molybdate Titration for Lead.*

Weight of Pb taken.	Weight of $CaSO_4$ added.	c.c. Molybdate Solution required.	Lead calculated, per Cent.
0·3415 grm. ...	nil	29·0	100·0
" " ...	0·0036	29·1	100·3
" " ...	0·0073	29·2	100·7
" " ...	0·0109	29·5	101·7
" " ...	0·0146	29·8	102·7
" " ...	0·0183	29·8	102·7
" " ...	0·0256	29·8	102·7
" " ...	0·0292	29·9	103·1
" " ...	0·0327	29·9	103·1
" " ...	0·0366	30·0	103·4
" " ...	0·0402	30·2	104·1
" " ...	0·0439	30·7	105·8
" " ...	0·0475	30·7	105·8
" " ...	0·0512	30·7	105·8

On slightly altering the conditions of the experiments—as, for example, by using more dilute solutions or by varying the amounts of reagents used—the results obtained were found to vary considerably. The following table will illustrate this point; the determinations were carried out in exactly the same manner as the previous set, but a smaller quantity of lead was taken.

TABLE II.—*Effect of Larger Quantities of* $CaSO_4$ *on the Ammonium Molybdate Titration for Lead.*

Weight of Pb taken.	Weight of $CaSO_4$ added.	c.c. Molybdate Solution required.	Lead calculated, per Cent.
0·2005 grm. ...	nil	18·7	100·0
" " ...	0·0549	19·4	103·7
" " ...	0·0612	19·6	104·8
" " ...	0·0658	19·6	104·8
" " ...	0·0695	19·6	104·8
" " ...	0·0732	19·6	104·8
" " ...	0·0805	19·6	104·8
" " ...	0·0842	19·7	105·3
" " ...	0·0915	19·7	105·3
" " ...	0·0951	19·8	105·9
" " ...	0·1134	19·9	106·4
" " ...	0·1317	19·9	106·4
" " ...	0·1500	20·0	106·9
" " ...	0·1685	20·0	106·9
" " ...	0·1866	20·0	106·9
" " ...	0·2049	20·2	108·0
" " ...	0·2232	20·2	108·0
" " ...	0·2415	20·4	109·1

It will be seen, on comparing these tables, that mass action plays a considerable part in these reactions; for in the case of Table II., in which the lead solution is more dilute, 0·0915 grm. calcium sulphate (45·6 per cent.) interferes to a less extent than 0·0512 grm. calcium sulphate (15 per cent.) does in the results given in Table I.

The table given below shows the effect of larger quantities of calcium sulphate on the assay. In this case separate weights of the sulphate and of the calcium sulphate were taken, the same amount of ammonium acetate was used in each case, and the whole conditions of the experiment were kept identical.

TABLE III.

Weight of Pb taken.	$CaSO_4$ taken. Grms.	$CaSO_4$ per Cent. of $PbSO_4$ present.	c.c. Ammonium Molybdate required.	Lead calculated, per Cent.
0·25 grm. ...	—	—	23·7	100·0
,, ,, ...	0·055	15	25·1	105·9
,, ,, ...	0·110	30	26·1	110·1
,, ,, ...	0·183	50	26·7	112·6
,, ,, ...	0·256	70	27·1	114·3
,, ,, ...	0·329	90	27·1	114·3
,, ,, ...	0·366	100	27·1	114·3
,, ,, ...	0·549	150	27·1	114·3

From the results given it will be seen that calcium sulphate, even in small quantities, causes high results to be obtained for lead; but from Table III. it will also be seen that the range of this interference is limited to a certain amount, above which no further increase in the lead results is obtained.

When the conditions of the experiments are not kept absolutely constant, the presence of calcium sulphate raises the results in an irregular manner, as stated by Low (*loc. cit.*); but when the conditions are kept the same, then additions of calcium sulphate cause systematic increases in the calculated amount of lead present. There are indications that double molybdates are precipitated when calcium sulphate is present; for on calculating from the molybdate used the composition of the precipitate formed in the case of the results given in Table III., the following results are found:

$CaSO_4$ present.	Calculated Formula of Precipitate.
15 per cent. of the $PbSO_4$ present.	17 $PbMoO_4$, $CaMoO_4$.
30 ,, ,, ,,	10 ,, ,,
50 ,, ,, ,,	8 ,, ,,
70 to 150 ,, ,, ,,	7 ,, ,,

The results of similar experiments in which a solution of lime in ammonium acetate was used in place of the calcium sulphate solution are given in Table IV., and in this case a more dilute solution of ammonium molybdate was used, to insure greater accuracy, the first 100 c.c. in each case being added by means of a Stas pipette.

TABLE IV.—*Effect of Lime on the Ammonium Molybdate Titration for Lead.*

Weight of Pb taken.	CaO added. Grms.	CaO per Cent. of $PbSO_4$ present.	c.c. Ammonium Molybdate required.	Lead calculated, per Cent.
0·3074 grm. ...	—	—	106·5	100·0
„ „ ...	0·0225	5	108·3	101·7
„ „ ...	0·0450	10	109·1	102·4
„ „ ...	0·0675	15	109·9	103·2
„ „ ...	0·0900	20	110·7	103·9
„ „ ...	0·1350	30	111·8	105·0
„ „ ...	0·2250	50	113·0	106·1
„ „ ...	0·4500	100	114·6	107·6
„ „ ...	0·6750	150	115·4	108·3

From these figures it is seen that lime acts in a similar manner to calcium sulphate in causing high results to be obtained for lead when the lime is present in the ammonium acetate solution of the lead sulphate. On carrying out a large number of experiments with lime, under somewhat varying conditions as to strength of solution, etc., the same irregularities as to the amount of interference introduced were noticed.

Similar experiments carried out with barium and strontium sulphates show that these act in quite a different manner; for by boiling mixtures of these sulphates with sulphate of lead in ammonium acetate solution somewhat low results were obtained in every case, owing to these insoluble sulphates holding small quantities of lead sulphate, and thus preventing the complete solution of the lead.

From this work the following conclusions may be drawn: (1) That lime or calcium sulphate in solution in ammonium acetate does not form an insoluble molybdate when boiled with ammonium molybdate solution. (2) That lime or calcium sulphate in ammonium acetate solution does not react with precipitated lead molybdate. (3) That when lime or calcium sulphate is present with lead sulphate in ammonium acetate solution, then, on boiling with ammonium molybdate solution, the insoluble molybdate formed contains both lead and lime. (4) From conclusion (3) it follows that it is impossible to determine lead sulphate direct by solution in ammonium acetate when lime or calcium sulphate is present; and (5) in the ordinary lead assay for materials containing lime, etc., it is necessary to separate the lead, or else to take special precautions to wash the lead sulphate free from calcium sulphate before proceeding with the solution of the lead sulphate. (6) Barium and strontium sulphates do not act in the same manner as calcium sulphate, and tend to give low results instead of high results.

METALLURGY DEPARTMENT,
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DISCUSSION.

Mr. RICHMOND asked whether the authors had any further data as to the composition of these double molybdates beyond the excess of molybdate used. Had the double molybdate been actually separated, and its composition determined? Looking at the figures, it seemed to him that by no means all the calcium had been precipitated, and that the proportion that was precipitated was not the same in every case. He should like also to ask whether the authors could give the reason why there was no further increase in the percentage of lead found when the calcium sulphate exceeded 70 per cent. Was it due to the fact that the solution was saturated, and that the excess of calcium sulphate was not in solution? Or was it due to the fact that under the conditions of mass action an equilibrium was approached at the point where the maximum figure was obtained?

Mr. RAYMOND ROSS asked whether any of the impurities referred to were likely to be found in the finished lead—*i.e.*, in ordinary commercial lead.

Mr. BANNISTER, in reply, said that the actual composition of the double molybdates had not been ascertained; it would be seen from the paper that the readings obtained were merely considered to be an indication that double molybdates were formed. It was, however, curious that the results of a whole set of experiments should calculate out almost exactly in molecular proportions. They could not explain why the increase in the figures for lead stopped when 70 per cent. of calcium sulphate was reached. The saturation-point, however, was certainly not reached there, for even with as much as 150 per cent. of calcium sulphate the whole of this went into solution before the titration was commenced. These impurities were present in many lead ores, and also in lead slags and other furnace products, and it was important that the possibility of interference should be recognised in commercial work.

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THE DETECTION OF HEAVY PETROLEUM IN PAINTS AND VEGETABLE OILS.

BY W. B. POLLARD.

(*Read at the Meeting, May* 1, 1912.)

OWING to the present extensive use of turpentine substitutes, petroleum is now frequently found in mixed paints. Whatever views are held with regard to the value of light petroleum as a turpentine substitute, there is no doubt that heavy petroleum should never be present in paints. As the drying property of such paints can never be satisfactory, it is important to be able to detect the presence of this substance. Light petroleum can be readily separated by steam distillation; and by the action of sulphuric acid, this may further be separated from any turpentine present. Heavy petroleum is not so readily detected, though it can be found by the method described in Allen's "Commercial Organic Analysis." This method, though satisfactory for quantitative work, requires far too much time and manipulation to be of much use as a qualitative test for petroleum. Experiments were therefore made to find in what way it could be modified, in order to give rapid results. It was noticed that when linseed oil is poured into an excess of molten caustic soda and stirred with an iron rod, complete saponification takes place in a few seconds; the soap thus obtained, after cooling, can be extracted with petroleum ether without any passing into the solution and without any emulsion being formed. The petroleum ether extract, which can easily be filtered, will then contain the unsaponifiable oil. Should heavy petroleum be present, it will often betray its presence by the strong fluorescence imparted to the solution.

On evaporating the petroleum ether, petroleum is left behind, and a further examination can be made. In the case of normal linseed oils, a small residue of unsaponifiable oil, rarely exceeding 2 per cent., is always left after extraction; it was noticed that this unsaponifiable oil could further be removed by boiling with a good excess of strong nitric acid, only the minutest trace, if any, remaining after this treatment. Should, however, heavy petroleum be present in the linseed oil, this will reveal itself by the large amount of unsaponifiable matter, and will remain unattacked by the treatment with nitric acid. It was found that as little as 0·5 per cent. of heavy petroleum, when present in linseed oil, could be detected by this means. It is most conspicuous when the nitric acid is warmed, as the oil tends to creep up the side of the tube. Light petroleum or turpentine is not detected in this way, as they volatilise at the temperature at which the saponification takes place; as previously mentioned, they would be detected by steam distillation.

The actual method of conducting the test is as follows:—About 10 grms. of caustic soda are placed in a nickel crucible of 60 to 100 c.c. capacity, and fused over a Bunsen burner. When all the soda has just melted, the crucible is removed from the flame, and about 5 c.c. of the oil or paint quickly poured in. The contents are well stirred with an iron rod, and the crucible allowed to stand in a little water to cool. When cool, about 50 c.c. of petroleum ether are poured into the crucible, and the contents well stirred with the iron rod. By this method the soap forms a powder which settles at the bottom of the crucible, and an emulsion is never produced.

The petroleum ether solution is decanted through a filter-paper and received in a test-tube. It is then boiled off, the vapour being conducted away through a glass tube fixed by means of a cork in the mouth of the test-tube. The residue can now be further examined; this is done by boiling with about 3 c.c. of nitric acid, when any residue beyond a minute trace will be petroleum. Should a paint be found by this method to contain petroleum, it must be absolutely condemned, as its drying properties can never be satisfactory.

It is sometimes stated that resin oil is added to paints. Should this occur, it would be detected by the large amount of unsaponifiable matter, but would be distinguished from petroleum by the fact that it is oxidised by boiling with nitric acid. It might be thought that the Zeiss butyro-refractometer reading of the unsaponifiable matter would afford an indication of the presence of petroleum; this, however, is not the case, as both the heavy petroleum and the unsaponifiable matter normally present in linseed oil give readings which are outside the scale of the instrument.

This method may be used in examining other kinds of saponifiable oils for petroleum adulteration. A small quantity of unsaponifiable oil is left in nearly every case, but on boiling this with an excess of nitric acid, it is invariably destroyed.

The method can also be applied in the examination of stearine candles, which, when they contain paraffin, soften at a temperature considerably below their melting-point.

No claim is made that the method is quantitative, but it affords a ready means for the detection of heavy petroleums when mixed with saponifiable oil, and should not take more than about ten minutes to perform.

Survey Department Laboratories, Cairo.

Discussion.

Mr. **Raymond Ross** said that a determination of the factor $\frac{\mu-1}{d}$ in the residue would nearly always indicate whether it consisted of petroleum, and would in many cases show to what class of petroleum it belonged. In the case of nearly all petroleum oils this value was quite definite, varying from 0·557 to 0·543, while the figure for fatty oils was much different.

The **President** agreed that it seemed improbable that so little as 1 per cent. of mineral oil would be used as an actual adulterant, unless it were added to mask some other addition. Small quantities of mineral oil were, however, sometimes met with in fatty oils obtained by extraction with a solvent, owing to incomplete evaporation of the solvent. This had the effect of considerably lowering the flash-point of the oil, and also, to some extent, the viscosity.

Mr. **E. Richards Bolton** said that it was now unusual to find any trace of solvent in extracted oil, because in most modern extraction plants arrangements were made for blowing steam through the oil in order to remove the last traces of solvent, and manufacturers, for reasons of economy, were usually careful to carry out this operation as thoroughly as possible.

The **President** said that, although it might occur but rarely, it certainly was occasionally the case that the manufacturer did not succeed in removing the last traces of volatile solvent. A sample of extracted rape oil that he had lately examined lost 0·36 per cent. when 1 grm. was heated for an hour in the water-oven, and its flash-point (close test) was only 286° F., whereas the flash-point of rape oil obtained by pressure from the seed would not be below 400° F.

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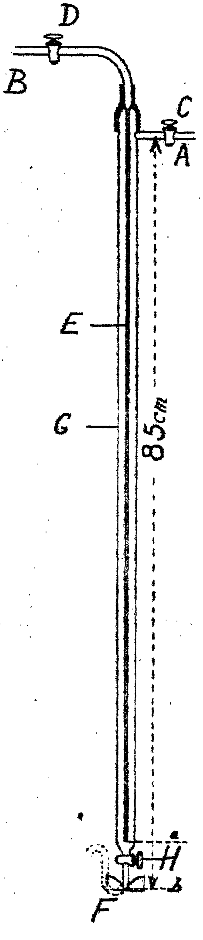
A CONVENIENT APPARATUS FOR OBTAINING AN AVERAGE SAMPLE OF GAS, AND FOR REGULATING THE FLOW OF A GAS INTO AN EVACUATED VESSEL.

By FRANK STURDY SINNATT.

(*Read at the Meeting, April 3, 1912.*)

The apparatus was devised to permit of the accurate regulation of the flow of gas into an evacuated vessel. The rate at which the gas is collected in the exhausted vessel is uniform within certain limits, and the pressure at which the gas is removed from any source is constant, but may be altered if required. After a preliminary adjustment little attention is necessary.



The figure shows a form of the apparatus constructed of glass, and the dimensions mentioned are those used for an apparatus for small volumes of gas (about 1 litre). A long burette makes a convenient main tube *G*, but the diameter may be less than that of the usual burette, if the volume of gas to be obtained is small. By this means an economy in the mercury necessary for the apparatus may be effected. The capillary tube *E* should be about 1 to 1·5 mm. in bore, and the walls should be of stout glass, in order to withstand the vibration which takes place in the tube. The volume of the capillary tube depends upon the diameter of the main tube. The end of the capillary tube is brought as close to the bottom of the main tube as possible, providing the flow of mercury from the latter is not interfered with. The exit of the main tube should be short, or bent into a seal, as indicated by the broken line. The reservoir *F* may be replaced by a levelling bottle during the evacuation of the apparatus, but this must be removed before the actual working of the apparatus is started. The vessel in which the gas is to be collected—*e.g.*, an ordinary gas sampler—is attached at *A* by pressure tubing, and by a second opening connected to a vacuum pump. The apparatus may be used in the following manner: The tap *D* is closed, and the whole exhausted, mercury being allowed to enter the main tube from the reservoir *F*. When the mercury level has reached the required point in the main tube, the sampling vessel is closed to the vacuum pump, and then disconnected. The tap *H* is then closed. The sample of gas is obtained by connecting the gas supply to *B*, and opening the tap *D* slowly; owing to the difference in pressure between the gas vessel and the capillary tube, gas is drawn down the capillary tube, and passes through the column of mercury into the gas vessel. If the distance between *a* and *b* be great, the tap should be opened cautiously; the less this distance the better, so long as the diminished pressure is sufficient to overcome that of the gas to be sampled. When the diminished pressure in the gas vessel has reached that represented by the height of the mercury column from *a* to the surface of the mercury in the main tube, gas no longer enters the gas vessel. The tap *D* may now be opened fully. Mercury is then allowed to flow from the main tube, drop by drop, into the reservoir. As the mercury leaves the tube, gas enters the gas vessel. The rate at which the mercury is allowed to flow determines the rate of entry of the gas into the gas-sampling vessel. When the tap is set, the rate at which the mercury falls into the reservoir is constant, as it depends upon the height of the column of mercury *a* to *b*; this flow is constant until the end of the capillary tube no longer dips into mercury. The distance *a* to *b* may be altered by moving the capillary tube, and consequently the gas may be drawn from a source at any diminished pressure required. By arranging for the mercury to run from the tube in a certain time, a representative sample of the gas throughout that period is collected in the gas vessel. It will be seen that the volume of the sample of gas collected is independent of the size of the apparatus itself. The condition of the gas is comparable with that of gas which has been collected in vessels previously filled with dry mercury.

The use of mercury in aspirators of more than 1 litre capacity is generally considered to be expensive, and too cumbersome for practical purposes. In this respect it is hoped the apparatus may be of some practical use.

An efficient water-pump serves for many purposes for exhausting the apparatus, and by washing the whole apparatus through with the gas to be sampled, the error due to the presence of air may be eliminated. If the pressure in the apparatus be noted, and the volume of the sampling vessel is known, the volume of gas entering the vessel may be calculated.

It is essential that the temperature of the vessel in which the gas is being collected should be as constant as possible throughout the period of sampling, in order to prevent any variation in pressure from that cause. It will be found advantageous to arrange the capillary tube to touch the wall of the main tube throughout its whole length. Instead of the gas passing through the column of mercury in irregular bubbles, and splashing at the surface, it passes up the canal formed at the two glass surfaces in a continuous and regular stream. The passage of the gas through the column of mercury is then scarcely perceptible, being accompanied by a slight vibration in the tube and mercury column.

An alternative way of using the apparatus is to fill the main tube, connections, and capillary tube with mercury, by means of a levelling tube, and then attach a gas vessel which has been previously evacuated at *A*, and proceed in the manner described for admitting the gas.

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THE USE OF METHYLENE BLUE AS AN INDICATOR IN IODIMETRIC TITRATIONS.

BY FRANK STURDY SINNATT.

(*Read at the Meeting, June* 5, 1912.)

IN a previous publication (ANALYST, 1910, 35, 309) it was shown that a dilute aqueous solution of methylene blue may be used in place of starch as an indicator in iodimetric titrations. The determinations quoted in that publication were made with a solution of methylene blue prepared by dissolving pure methylene blue in water. It has since been found that the usual commercial forms of methylene blue may also be used without the delicacy of the colour change at the completion of the titration being interfered with. The solutions should be of a similar concentration to that suggested for the pure methylene blue—viz., 0·05 grm. per litre, and about 1 c.c. be taken for a titration. The following observations may be added to the previous note:

It will be found that the compound of iodine and methylene blue frequently separates from the solution as a precipitate. This occurs when an excessive amount of methylene blue has been added, but it will be found not to interfere with the observance of the colour change. There are no objections, such as hold in the case of starch, to the introduction of the methylene blue at the beginning of the titration, and no retardation of the change in colour has been observed.

Titrations have been made in order to determine the influence of inorganic compounds upon the behaviour of the indicator, and neutral salts and the following acids —acetic, hydrochloric, or sulphuric—have been found not to affect the colour change.

When a solution of iodine in alcohol or in aqueous potassium iodide solution is added to methylene blue dissolved in alcohol, the formation of the iodine compound of methylene blue is accompanied by the production of a green-coloured solution. The change in colour is delicate, and, although not very pronounced, is more easily observed than when no indicator at all is used. The addition of a reducing agent such as sodium thiosulphate to the green solution restores the colour of the methylene blue. It is suggested that methylene blue may be of use for the determination of iodine in the presence of alcohol, where it would be impossible to use another indicator.

It has been shown by Harrison (*Koll. Zeit.*, 1911, 9, 5) that the formation of the blue coloration when iodine solution is added to starch solution is interfered with by the introduction of ethyl alcohol. The volume of ethyl alcohol which prevents the formation of the blue coloration varies with the concentrations of the solutions, and in the experiments quoted the blue coloration was not definitely produced when 30 per cent. of alcohol by volume was present in the liquid. Before this percentage of alcohol is reached the formation of the blue compound is too indefinite to be of value as an indicator.

In order to test the value of methylene blue as an indicator in the presence of alcohol, the following determinations were carried out. The solutions used were sodium thiosulphate solution (1 c.c. = 0·01309 grm. iodine) made according to the usual directions, and starch solution made from pure potato starch. The ethyl alcohol contained 96 per cent. of alcohol by volume. In carrying out the check titrations with starch solution, sufficient water was added to lower the percentage of alcohol present to about 2 per cent. Generally three titrations were made for each experiment.

$\frac{N}{10}$ iodine solution (potassium iodide) was mixed with alcohol, 1 c.c. of methylene blue in alcoholic solution added, and the liquid titrated with sodium thiosulphate solution.

Indicator.	$\frac{N}{10}$ Iodine Solution.	Alcohol.	$\frac{N}{10}$ Sodium Thiosulphate required.
	c.c.	c.c.	c.c.
Methylene blue ...	10	20	9·7
" " ...	10	50	9·7
" " ...	10	100	9·7
Starch	10	—	9·7

The introduction of chloroform or carbon tetrachloride does not affect the titration if the percentage of alcohol present is sufficient to prevent the organic solvent separating from the solution.

Indicator.	$\frac{N}{10}$ Iodine.	Carbon Tetrachloride.	Chloroform.	Alcohol.	$\frac{N}{10}$ Sodium Thiosulphate required.
	c.c.	c.c.	c.c.	c.c.	c.c.
Methylene blue ...	10	10	—	100	9·7
" " ...	10	—	10	100	9·7
Starch	10	10	—	—	9·7
"	10	—	10	—	9·7

A solution of iodine in alcohol was prepared, having a concentration approximately decinormal; no potassium iodide was used.

Indicator.	$\frac{N}{10}$ Iodine.	Chloroform.	Carbon Tetrachloride.	Alcohol.	$\frac{N}{10}$ Sodium Thiosulphate required.
	c.c.	c.c.	c.c.	c.c.	c.c.
Methylene blue ...	10	—	—	—	12·15
" " ..	10	—	—	50	12·20
" " ...	10	—	—	100	12·20
" " ...	10	10	—	100	12·15
" " ...	10	—	10	100	12·20
Starch	10	—	—	—	12·15

Before the check titration, in which starch was used as the indicator, was performed, potassium iodide solution was added to the alcoholic solution of iodine, and then a large volume of water, and the titration carried on in the usual manner.

For Wijs' solution an aqueous solution of potassium iodide was added, then alcohol until the whole formed a clear solution.

Indicator.	Wijs' Solution.	Carbon Tetrachloride.	Chloroform.	Potassium Iodide.	Alcohol.	$\frac{N}{10}$ Sodium Thiosulphate required.
	c.c.	c.c.	c.c.	c.c.	c.c.	c.c.
Methylene blue	10	—	—	20	100	19·6
" "	10	—	10	20	100	19·6
" "	10	10	—	20	100	19·6
Starch ...	10	—	—	20	—	19·6

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

ANALYSIS OF FOOD AND DRUGS.

Amounts of Arsenic in Vegetable Foods. F. Jadin and A. Astruc. (*Comptes. rend.*, 1912, **154**, 893-896.)—Estimations were made, by a modification of Marsh's method, of the amounts of arsenic in thirty-six samples of fresh and dried vegetables and fruits, typical of all those of most common occurrence. In the fresh vegetables the proportion of arsenic ranged from 0·004 mgrm. (peas) to 0·023 mgrm. (lettuce) per 100 grms. The edible portion of apples contained 0·005, and that of oranges 0·011 mgrm. per 100 grms.; whilst in the case of dried fruits and vegetables the amounts varied from 0·007 (rice) to 0·025 (almonds and red haricot beans) mgrm.

C. A. M.

Detection of Small Quantities of Chloral in Presence of Chloroform. T. Jona. (*Giorn. Farm. Chim.*, 1912, **61**, 57; through *Chem. Zentralbl.*, 1912, I., 1148.)—Chloral is usually detected by the reducing action of the formic acid into which it is first converted. This method is inapplicable in presence of compounds containing the group CCl_3, such as chloroform and trichloracetic acid, whilst bromoform and iodoform may also give rise to illusive results. Chloral cannot be separated from chloroform by distillation. The following method is recommended: In a flask closed by a plug of cotton-wool, the substance, dissolved in water and slightly acidulated with sulphuric acid, is allowed to stand, without warming, in contact with zinc until the evolution of hydrogen has ceased; a strip of filter-paper, soaked in a fresh solution of sodium nitroprusside and 5 per cent. piperidine solution, is hung in the neck of the flask, and the liquid warmed, but not allowed to reach the paper. The acetaldehyde formed by reduction of the chloral colours the paper blue. In this manner 1 part of chloral hydrate in 20,000 can be detected. O. E. M.

Method for the Determination of the "Gasoline Colour Value" of Flour. A. L. Winton.

(*Bull.* 137, *Bureau of Chem., U.S. Dept. of Agriculture.*)—Winton and Shanley (*U.S. Dept. of Agriculture, Bull.* 122, p. 217) have suggested a qualitative gasoline test for bleaching, which corroborates the nitrite test, but it does not give quantitative results. The method described is of value in the comparison of patent or other high-grade flours which owe their colour to the kind of wheat, not to mechanical impurities, as a means of detecting bleaching. Only the colour soluble in oil is measured, and not the total colour, or the colour due to mechanical impurities. Twenty grms. of the flour are placed in a wide-mouthed glass-stoppered bottle, and 100 c.c. of colourless gasoline are added. The bottle is shaken vigorously for five minutes, and is then allowed to stand for sixteen hours, when it is again shaken, and the contents filtered through a dry paper into an Erlenmeyer flask, keeping the funnel covered with a watch-glass. The colour value of the clear solution is determined in a Schreiner colorimeter, using for comparison a 0·005 per cent. aqueous solution of potassium chromate; this solution corresponds to a gasoline number of 1. The colorimeter tube containing the gasoline solution should be adjusted so as to read 50 mm., and the tube containing the standard chromate solution is raised or lowered until the shades of yellow in both tubes match. The reading of the chromate solution, divided by that of the gasoline solution, gives the gasoline colour value. If desired, the colour value may be determined in Nessler tubes, using for comparison chromate solutions of various dilutions, and filling the tubes in all cases to the height of 50 mm. A number of determinations are given, showing the effect of variety, grade, ageing, and bleaching, on the gasoline value. Bleaching with nitrogen peroxide (2 parts of nitrous nitrogen per million) immediately produced about the same degree of whitening as ageing for thirty weeks. Ageing of the bleached flour brought about further bleaching, so that after thirty weeks hardly any colour remained. If the flour is known to have been milled within ten weeks, the gasoline colour value may be depended upon to corroborate the determination of nitrous nitrogen as a means of detecting bleaching. H. F. E. H.

Composition of Hare Fat. J. Klimont.

(*Monatsh. Chem.*, 1912, **33**, 441-446.)—A specimen of this fat yielded the following chemical and physical constants:

Sp. gr. at 50° C.	0·9134
Melting-point	35° C.
Solidifying-point	29° C.
Acid value	15·3
Saponification value	204·9
Iodine value	107·0

The fat was found to consist principally of tripalmitin mixed with glycerides of unsaturated, fluid fatty acids; the latter, however, did not contain linoleic acid to any great extent. W. P. S.

Saponificaton of Fats by Sulphuric Acid. A. Grün and O. Corelli. (*Zeitsch. angew. Chem.*, 1912, **25**, 665-670.)—All the experiments carried out by the authors on the action of sulphuric acid on pure triglycerides of the higher fatty acids yielded the same result—namely, the formation of diglycerides. On treating tripalmitin for several hours at 70° C. with a tenfold molecular quantity of sulphuric acid of sp. gr. 1·845, the resulting mixture contained 56·5 per cent. of free palmitic acid and 43·5 per cent. of neutral fat, the latter consisting of 30 per cent. of unaltered tripalmitin and 70 per cent. of dipalmitin. On doubling the quantity of sulphuric acid, the product consisted of 65 per cent. of free acid and 35 per cent. of neutral glyceride, the latter being 25 per cent. unaltered glyceride and 75 per cent. dipalmitin. Tristearin yielded approximately similar results. The reaction appears to proceed beyond the diglyceride stage, as is shown by the proportion of free fatty acid to neutral fat, and, although monoglycerides could not be detected at any stage of the reaction, it is possible that monoglycerides are formed and immediately split up into fatty acids and glycerol or glycerol esters of sulphuric acid. W. P. S.

Mkonga Oil. W. Arnold. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 391-392.)—The seeds of the Mkonga-tree (*Balamites ægyptica*), which grows in German East Africa, yield a yellow oil having a nutty taste and odour. A specimen of the oil yielded the following figures on analysis:

	Oil.	Non-Volatile Fatty Acids.
Sp. gr. at 15° C.	0·9173	—
$[N]_D$ 40° C.	55·9	—
Saponification value	195·6	206·1
Reichert-Meissl value	0·55	—
Polenske value	0·40	0·50
Iodine value	77·2	82·9
Acid value	8·5	—
Solidifying-point	Turbid at 8·8° C.	—
Stearic acid	2·4 per cent.	2·6 per cent.
Unsaponifiable matter	0·07 ,, ,,	—

The oil yielded negative reactions when tested for cottonseed and sesame oils. W. P. S.

Sources of Error in the Estimation of the Polenske Value of Fats. W. Arnold. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 389-391.)—From the results of analyses of fats which have been published from time to time, it is evident that incorrect results are sometimes obtained in the estimation of the Polenske value, and the author draws attention to the errors in manipulation which tend to cause these discrepancies. Many of these points have been mentioned already (*cf.* ANALYST, 1904, **29**, 154; 1909, **34**, 223, etc.). The distillation flask should be of 300 c.c. capacity, and all the dimensions of the apparatus described originally by Polenske should be adhered to; the use of wire gauze under the flask should be avoided, but the flask should rest on an asbestos ring, the hole in which is 6·5 cm. in diameter. The flame of the burner should be sufficiently large to cover the bottom of the flask, but should not make the edges of the asbestos ring incandescent. Powdered pumice must be employed instead of pieces of pumice to prevent "bumping," and the distillate should measure exactly 110 c.c. As the Polenske value varies with the quantity of glycerol used in the saponification of the fat, care should be taken that exactly 20 grms. of glycerol be used for each estimation. In the author's opinion, the correct working of any apparatus is ascertained by making an estimation of the Polenske value of a specimen of pure, fresh lard; the value found should be approximately 0·5, and should never exceed 0·65. W. P. S.

Influence of the Concentration of Hydrogen Ions on the Baking Qualities of Flour. H. Jessen-Hansen. (*Comptes rend. Trav. Lab. de Carlsberg*, 1911, **10**, 170-206.)—Determinations of the concentration of hydrogen ions were made with the object of ascertaining the effect of the addition of acid substances to flour, the methods employed for the measurement being the conductivity and colorimetric processes described previously by the author (ANALYST, 1909, **34**, 498). It was found that for dough prepared from wheaten flour there is a certain definite concentration of hydrogen ions at which the best baking results are obtained, and that this concentration is higher than in dough prepared from freshly milled flour and distilled water or fresh milk. The optimum concentration corresponds approximately with the exponent $p_{\text{H}}^{+} = 5$; in the case of the best qualities of flour it may be a little higher, and for ordinary flours slightly lower. The flour from the central portion of the wheat grains has a greater acidity—that is, a higher concentration—than the outer portion, the bran having the least. The addition of "improvers" to flour has scarcely any other effect than to increase the concentration of the hydrogen ions. Processes for the estimation of the gluten, which depend on the separation of the gluten by washing the flour, do not afford trustworthy data as to the baking qualities of flours. W. P. S.

Incineration of Food-Stuffs and other Organic Substances prior to the Determination of their Phosphorus-Content. A. Vozárik. (*Zeitsch. physiol. Chem.*, 1912, **76**, 426-432; through *J. Soc. Chem. Ind.*, 1912, **31**, 250.)—The phosphorus in food-stuffs is in part present in the form of phosphatides, which are more or less volatile below 100° C. For the incineration of such products, as a first step in the determination of the phosphorus, the author proposes the following modification of Eschka's method: One grm. of the finely-ground sample if of animal origin, or 3 grms. if vegetable, is well mixed with 0·2 grm. of magnesia in a platinum crucible. The magnesia employed should be as light and voluminous as possible, and free from lumps. The open crucible is laid obliquely, and at first warmed carefully near the edge until all volatile matters have been expelled. The burner is then moved towards the base of the crucible, the cover is placed obliquely in the mouth, and the heating is continued until incineration is complete. The whole operation may require half an hour to two hours in the case of animal matters, and two to three and a half hours with vegetable matters. The ash is obtained as a powder, which does not adhere to the inside of the crucible.

Determination of Phosphorus in Food-Stuffs and other Organic Substances by Titration with Uranyl Solutions. A. Vozárik. (*Zeitsch. physiol. Chem.*, 1912, **76**, 433-456; through *J. Soc. Chem. Ind.*, 1912, **31**, 250.)—In the determination of phosphorus in incinerated organic products (see preceding abstract) by titration with uranyl nitrate solutions, errors may arise from the presence of mineral acids, large quantities of acetic acid, various dissolved salts, especially the acetates of the alkali and alkaline earth-metals, and the phosphates of iron and aluminium. The liquid to be titrated should contain no phosphates acid towards phenolphthaleïn and no aluminium or iron phosphate. The disturbing influence of free acetic acid begins to be appreciable at a concentration of 0·36 per cent. when cochineal is used as indicator, and 0·6 per cent. when potassium ferrocyanide is used. Errors are introduced by sodium, ammonium, and magnesium acetates at concentrations exceeding 4, 2, and 0·75 per cent. respectively. The presence of strong acids leads to high results with cochineal as indicator, but to low results with ferrocyanide. If both are used at the same time, the divergence of the results gives an indication of the magnitude of the error. When the liquid titrated contains a large quantity of foreign salts, the influence of the latter on the result must be determined and a corresponding correction made. A certain small volume of uranyl solution is required to produce the indicator reaction. If allowance is made for this, there is strict proportionality between the volume of uranyl solution required and the quantity of phosphorus present, irrespective of the concentration of the latter. The author gives full details for carrying out the determination of phosphorus in incinerated organic substances by this method. The results obtained agree well with those found by the gravimetric citrate method.

Formic Acid in Honey. H. Fincke. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 255-267.)—That formic acid is not invariably present in honey is shown by the results of analyses of numerous samples of honey recorded by the author. Many of the samples were free from formic acid, but the majority contained traces of a reducing volatile acid (as estimated by the mercuric chloride method; *cf.* ANALYST, 1911, **36**, 103), the quantity of which did not generally exceed 0·003 per cent.; in a few cases the quantity amounted to 0·02 per cent., calculated as formic acid. The volatile acid was present partly in the free state and partly in the form of salts; esters of the acid could not be detected. In the course of the investigation it was found that invert sugar, prepared by heating cane sugar with acids, yields small quantities of formic acid when submitted to steam distillation; invert sugar prepared with invertase does not yield formic acid. W. P. S.

Kola Nuts from British West Africa. (*Bull. Imp. Inst.*, 1912, **10**, 34-37.)—The samples of "kola nuts" received during 1909-10 from the Gold Coast were found on examination to have the following composition:

	Moisture, per Cent.		Caffeine.			
			In Nuts as received, per Cent.		In Nuts after Drying at 100° C., per Cent.	
	1.	2.	1.	2.	1.	2.
Cola acuminata (genuine kola nuts)	50·16	15·2	0·73	1·7	1·5	2·1
C. Johnsoni (false kola nuts)...	49·32	14·8	0·67	0·7	1·3	0·8
C. verticillata „ „ ...	52·60	14·2	0·55	0·7	1·1	0·8

The so-called "bitter kola seeds" contain no caffeine, and are the product of *Garcinia Conrauana.* A. R. T.

Estimation of Dry Matter in Potatoes and the Applicability of the Ordinary Tables. H. Björn-Andersen. (*Zeitsch. anal. Chem.*, 1912, **51**, 341-359.)—From experiments of his own, as well as from consideration of the data on which Maercker and others have founded their tables, the author concludes that the sp. gr. of potatoes, however carefully determined, is a most uncertain guide to their content of dry matter. In at least one experiment in ten the error will exceed ± 1 per cent., which is far too large an error where the number to be estimated is 22·5 ± 3 per cent., which limits for dry substance would include most potatoes grown for their starch. If a table is to be used, it should rather be one connecting dry substance as determined by the most exact methods with the number obtained by some empirical, and not too slow or complicated, method of drying. The author disintegrates 4 or 5 kgrms. of potatoes in a mincing-machine with holes 0·9 cm. in diameter, and 2 kgrms. of the resulting paste is put through the machine twice more, using plates with holes 0·45 and 0·3 cm. in diameter. A 10-grm. sample is then dried for eight days at 99° to 100° C. If the drying be discontinued after twenty-four hours, the results are 0·4 ± 0·04 per cent. too high, and it is suggested that drying in a vigorously boiling, well-ventilated steam oven for twenty-four hours, with a deduction of 0·4 per cent. from the value found, would afford a good empirical method. G. C. J.

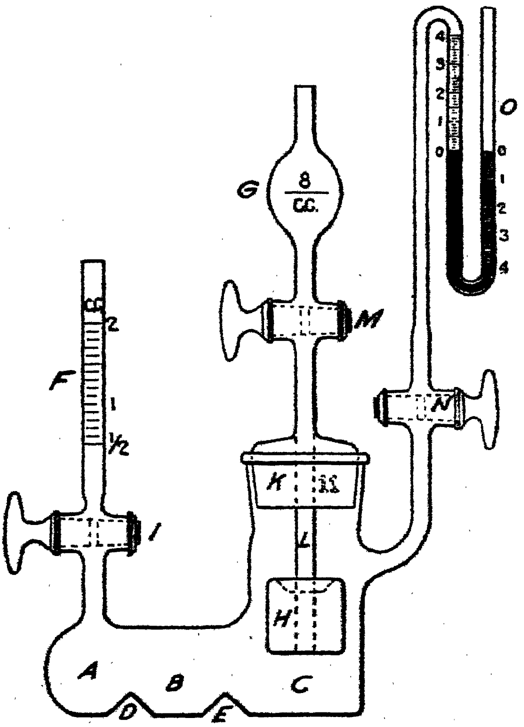
Detection of Adulteration of Cayenne Pepper. A. von Sigmond and M. Vuk. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 387-388.)—The authors have endeavoured to find a method for the detection of added oil in this condiment, but without success. As they have mentioned previously (*cf.* ANALYST, 1912, 56), the added foreign oil would remain on the surface of the particles, but attempts to separate this "surface" oil by pressing the pepper between filter-paper and extracting the oil absorbed by the latter by shaking the pepper with water and separating the oil by centrifugal action, etc., did not yield results which were of any use. The iodine value of the separated oil afforded no evidence of the presence of a foreign oil. W. P. S.

Estimation of the Total Solids of Vinegar. P. Lehmann and J. Gerum. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 267-274.)—Windisch and others have shown that the total solids of vinegar may be estimated correctly by evaporating the vinegar, treating the residue three times with water, and weighing the final residue; the latter may then be dissolved in a definite volume of water and the sp. gr. of the solution determined, a reference to tables giving the amount of total solids corresponding with the sp. gr. found. The authors now give an indirect method of estimating the total solids, which consists in estimating the quantity of acetic acid in the vinegar, multiplying the percentage quantity of this constituent by 0·0015, and subtracting the product from the sp. gr. of the vinegar. The difference is the sp. gr. of the solution of the total solids, and the quantity of the latter is then found by reference to tables. In case traces of alcohol or other substances lighter than water are present, 50 c.c. of the vinegar should be evaporated to 15 c.c., diluted to 50 c.c., and the sp. gr. determined. The solution is then titrated with $\frac{N}{1}$ potassium hydroxide solution, and the number of c.c. required for neutralisation is multiplied by 0·00018. The result, subtracted from the sp. gr., gives the sp. gr. of the solution of the total solids. W. P. S.

Estimation of Glycerol in Wine. S. Rothenfusser. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 332-337.)—In the method proposed the glycerol is converted into oxalic acid after the wine has been treated with stannous chloride to precipitate organic acids, and with ammoniacal basic lead acetate to remove sugars (*cf.* ANALYST, 1910, **35**, 208). When only a small quantity of sucrose is present in the wine, the addition of a somewhat large amount of the lead reagent effects its removal, provided that the casein solution is not employed; but if more than 1 per cent. of sucrose is present, the wine should be subjected to a preliminary heating with an acid in order to invert this sugar. The estimation is carried out as follows: A quantity of the wine is treated with an excess of sodium carbonate, and crystallised stannous chloride is then added until the mixture exhibits an acid reaction. The precipitated lactic and other organic acids are now removed by filtration, and the filtrate is diluted to a definite volume after the addition of ammoniacal basic lead acetate solution. The mixture is filtered, and an aliquot portion of the filtrate is treated with a small quantity of sodium phosphate and potassium carbonate, and evaporated to about one-third its volume. Powdered potassium permanganate is then added to the cold solution in excess, and after the lapse of thirty minutes the excess is destroyed by the addition of hydrogen peroxide. The oxalic acid formed by the oxidation of the glycerol is now precipitated as its calcium salt, which is collected, dissolved in diluted sulphuric acid, and titrated with permanganate solution in the usual way. Estimations of glycerol in artificial wines containing from 0·26 to 0·64 per cent. of glycerol yielded results which did not differ by more than 0·02 per cent. from the quantity present. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Measurement of the Oxidase Content of Plant Juices. H. H. Bunzel. (*J. Amer. Chem. Soc.*, 1912, **34**, 303.)—The oxidising enzymes in plants are of importance in connection with respiration and in many diseases; and in special cases they are concerned with the darkening of tea and of bread during its making, as well as with the hardening of the black Japanese lacquer prepared from the tree *Rhus vernicifera*. The author has elaborated a quantitative method involving the measurement of the amount of oxidation of pyrogallol by oxygen in the presence of the plant juice. Only fresh unfiltered juice was used. The apparatus (see Fig.) is clamped on the carriage of a shaking machine in a thermostat. Eight c.c. of fresh 1 per cent. pyrogallol solution are measured into the compartment *C* by means of the bulb *G*. Two c.c. of plant juice are measured into the compartment *A* from the burette *F*. The basket *H* is charged with 1 c.c. $\frac{N}{1}$ caustic soda solution. Only the stopcock *I* is left closed, and the interior of the thermostat is heated to the desired temperature. About 30 minutes later the windows of the thermostat are opened enough to allow the introduction of the arm, and the stopcock end closed. The shaking machine is then brought into action at an approximate rate of 5 complete movements in 3·3 seconds. At 15-minute intervals the shaking is interrupted long enough to read the manometers. The experiment is considered completed when successive readings are identical. The carbonic acid liberated can be approximately determined by lifting off the basket *H* at the ground joint *K*, adding a drop of phenolphthalein, and placing the whole in a special titration flask. In the experiments described, the pressure readings on the manometer are given, and the values reduced to the arbitrary volume of 150 c.c., this being about the capacity of the apparatus used. The absorption of oxygen comes to an end in about 2 hours, and the concentration of pyrogallol solution has no appreciable effect on the end result, provided it is over 0·2 and under 16 per cent. A certain quantity of potato juice is capable of bringing about the oxidation of a definite quantity of pyrogallol, any excess of which



remains unoxidised. The total oxygen absorption is at least approximately proportional to the quantity of potato juice present. Excess of caustic soda is necessary in the basket *H* to insure complete removal of the carbonic acid formed during the experiment. Care must be taken not to splash any pyrogallol into the alkali, or no satisfactory end result is obtained. Previous workers have shown that many enzymes lose their activity on vigorous shaking, and experiment showed that if the potato juice was shaken for 15 to 30 minutes before the addition of the oxidisable substance, its oxidising power was reduced to about half the original value; further shaking produces no effect. This indicates that two phases of the process are concerned, each of which may be measured separately, and to obtain the total oxidising effect of the plant juice measurements must be taken from the moment that shaking begins.

The author has applied his method to the investigation of the curly-top disease of sugar beets, and finds that the oxidase content is markedly greater in the diseased leaves, and shows wide variations, while normal leaves appear to be fairly constant. The deviation in oxidase content of the pathological leaves runs parallel with the appearance of the leaves. The main source of error in using the apparatus lies in the rise of pressure within it as soon as shaking is begun; but by allowing the apparatus and its contents to stay at the chosen temperature for at least half an hour, this may be almost entirely overcome. Within the limits of the experiments the amount of the chemical change is directly proportional to the concentration of the oxidase present, all other factors remaining the same. It thus appears that the behaviour of plant oxidase is contrary to the usual conception of enzyme action in general, since it would have been expected that small quantities of juice would bring about the oxidation of relatively large quantities of pyrogallol, and that the action would continue so long as pyrogallol and free oxygen are present; whereas the process comes to completion when only a small definite portion of the pyrogallol is oxidised, and while there is still an abundance of oxygen. It therefore seems that the oxidase in potato juice which accelerates the absorption of oxygen by pyrogallol is not an enzyme in the customary sense of the word, but is a substance entering directly into the reaction, and destroyed in the course of the same. As it is desirable to express the strength of the juice in terms of some standard, the author proposes as a unit for future experiments an oxidase solution of such strength that 1 litre of it will be capable of bringing about the consumption by pyrogallol of 8 grms. of oxygen. This unit of strength may not have any relation to the rate of the absorption, as it refers only to the total amount absorbed. It is left to future investigation to determine whether the strength of an oxidase solution, as expressed by this standard, is proportional to the rate at which the absorption takes place. The apparatus may be obtained from Machlett and Son, 143, East Twenty-third Street, New York City.

H. F. E. H.

Copper Fungicides. S. U. Pickering. (*J. Agric. Sci.*, 1912, **4**, 273-281.)—Gimingham and Barker (ANALYST, 1911, **36**, 351) questioned the validity of the principle that solubility of the copper compound is essential in order that it may exert its fungicidal action, and doubt that such solubility is brought about by the action of carbon dioxide, suggesting that mere contact between the fungus and an insoluble copper compound is sufficient to account for its action. It is doubtful whether the carbon dioxide in air can render soluble more than mere traces of copper in ordinary Bordeaux mixture. The author controverts these opinions, and cites further experimental work in support of his view that the efficacy of such fungicides depends upon the proportion of copper in them which is rendered soluble by the carbon dioxide of the air. The amount of copper rendered soluble may be estimated by immersing clean iron rods in the solution, removing them at intervals, wiping them free from copper, and noting their loss in weight. Solutions too dilute to show copper by the ferrocyanide method (unless previously concentrated) can thus be shown to contain copper. The greater the excess of lime present, the longer is the interval before the solution of copper commences; but in practical spraying this time is shortened, since the deposit dries up and isolates the particles of basic sulphate from those of lime. In spite of the small solubility of $10CuO,SO_3$, the greater part of the copper in it will pass into solution in a short time if any body be present, such as iron, to react with it and remove it from the solution; and this will occur to a much greater degree in the presence of such a large reacting surface as fungus spores or plant leaves. If fungicidal action depended, as Gimingham and Barker hold, on a solvent material exuded from the fungus, all copper compounds, or, at any rate, all the basic sulphates and carbonates, would be equally efficacious for a given weight of copper present, a proposition which is contrary to all experience. Ordinary Bordeaux mixture should be more effective than other compounds of the same copper content, for the lime itself appears to have a conspicuous fungicidal action, which action, the author claims, accounts for the results obtained by Gimingham and Barker (*loc. cit.*), when they found that ordinary Bordeaux mixture was affected in an atmosphere free from carbonic acid—a result which they accepted as proving that carbonic acid does not bring the copper of such a mixture into solution.

H. F. E. H.

Estimation of Sugar in Blood. E. Herzfeld. (*Zeitsch. physiol. Chem.*, 1912, 77, 420-424.)—The blood, or blood-serum, is treated with metaphosphoric acid to precipitate proteins; these are removed by filtration, and the filtrate, after the addition of potassium hydroxide, is titrated with a standardised methylene-blue solution. The latter is decolorised by aldoses, ketoses, and dextrins, in alkaline solution, and also by proteins, cholesterol, and bilirubin—hence the necessity of removing these by treatment with metaphosphoric acid; urea does not decolorise the methylene-blue solution. The standardisation of the methylene-blue solution is carried out by adding 0·5 c.c. of a 20 per cent. potassium hydroxide solution to an exactly measured quantity of a 0·1 per cent. dextrose solution, and heating the mixture nearly to boiling. When the hot solution begins to turn yellow in colour, a 0·001 per cent. aqueous methylene-blue solution is run in, drop by drop, until the colour is no longer discharged. Experiments carried out by the author show that 1 c.c. of the methylene-blue solution is equivalent to 0·00062 grm. of dextrose. In the case of blood, 5 c.c. of the sample are mixed with 15 c.c. of a 10 per cent. metaphosphoric acid solution, filtered after the lapse of ten minutes, and the precipitate washed with a small quantity of the metaphosphoric acid solution. The filtrate is then neutralised with potassium hydroxide, an excess of 0·5 c.c. of 20 per cent. potassium hydroxide solution is added, and the titration carried out as described above. The method yields concordant results. W. P. S.

Copper-Reducing Power of Normal Urine. N. Schulz. (*Zeitsch. physiol. Chem.*, 1912, **67**, 121-128.)—When normal urine, free from sugar, is treated with copper sulphate solution, and then rendered alkaline with an excess of sodium hydroxide solution, a clear blue solution is obtained, provided that too much copper has not been added; on heating the solution for some minutes, reduction of the copper takes place. If, however, the urine be rendered alkaline before the addition of the copper solution, a turbid solution is produced, and the copper is not reduced (Trommer's test for sugar). Creatinine solutions behave in a similar manner, the solution of the copper hydroxide or otherwise, depending on the order in which the reagents are added; as creatinine is converted into creatine by the action of alkalis, it is probable that this is the explanation of the reaction. W. P. S.

Method of Testing Water. Determination of the Power of Bacteria to Decompose Proteins. W. Spät. (*Arch. Hyg.*, 1912, **74**, 237-288; through *Chem. Zentralbl.*, 1912, I., 1332-1333.)—The method proposed consists in mixing quantities of the water under examination with varying amounts of a 2 per cent. sterilised peptone solution, maintaining the mixtures at 37° C., and estimating the ammonia formed. The activity of the bacteria as regards the production of ammonia is expressed in c.c. of $\frac{N}{100}$ solution. In the case of sterile waters, ammonia is not formed; with waters containing but few bacteria the production of ammonia is very small, but impure waters loaded with bacteria produce considerable quantities. The amount of ammonia formed is not always proportional to the number of bacteria present, as the true water bacteria are capable of producing but very small traces of ammonia, and practically the same may be said of pathogenic organisms and *B. coli*; but soil bacteria produce large quantities of ammonia, and the method mainly serves the purpose of detecting the presence of surface water in a supply, the introduction of which water indicates that other contaminating matters may find an entrance. At ordinary temperatures the production of ammonia is negligible, even in the case of contaminated waters, and samples may therefore be transported without being packed in ice. When a water-supply is examined periodically, any sudden increase in the quantity of ammonia obtained almost certainly points to the introduction of surface water. Sewage produces large quantities of ammonia, the decomposition of the peptone being accompanied by the formation of an extremely offensive odour, and, although there is a certain relation between the amount of ammonia produced and the quality of the sewage, some of the ammonia is lost during the incubation of the mixtures. The method will readily detect as little as 0·0005 c.c. sewage.

W. P. S.

ORGANIC ANALYSIS.

Aromatic Grass Oils. Part III. (*Bull. Imp. Inst.*, 1912, 10, 27-34—*cf.* ANALYST, 1912, 61; 197.)—New essential oils from species of *Cymbopogon* and from vetiver roots were found to give the following analytical results on examination:

	Cymbopogon Coloratus (Fiji). 3 Samples.	*Cymbopogon Polyneuros* (Ceylon). 4 Samples.	*Cymbopogon Sennaarensis* (Soudan). 1 Sample.
Sp. gr. 15°/15° C. ...	0·915 to 0·920	0·936 to 0·951	0·9383
Opt. rotation, 100 mm. at 20° C.	−7·7° (at 22° C.) to −8·7°	+30·9° to +55·25°	+34·25°
Aldehydes by sodium bisulphite, per cent.	42·0 to 43·5	—	(or ketones) 27
Citronellal, ,,	45·7; 49·5	—	—
Geraniol, ,,	15·6; 15·6	Total alcohols, 38·7 to 51·8	Total alcohols, 17·3
Solubility in—			
80 per cent. alcohol	1 : 1 or more vols.	1 : 1	—
70 ,, ,,	1 : 3; more alcohol gives cloudy solution.	Opalescence to turbidity with 1 : 10	—
Odour	Varying between citronella and lemon-grass oils.	Unlike citronella or lemon-grass oil.	Pennyroyal oil (pulegone present).

	Vetiver Oil (Fiji).	Oil from Seychelles Vetiver Roots.	Oil from Indian Vetiver Roots.
Sp. gr. 15°/15° C. ...	1·0298	1·0282	1·015 to 1·030
Opt. rotation, 100 mm. at 20° C.	—	+27·0°	+27·7°
Acid value	—	55·9	—
Saponification value	35·3	67·3	60 to 80
Solubility in 80 per cent. alcohol.	1 : $2\frac{1}{2}$; more alcohol gives cloudy solution.	1 : 1 or more.	1 : $1\frac{1}{2}$ or 2; more alcohol gives cloudy solution.

A. R. T.

Estimation of Acetaldehyde in Paraldehyde. **R. Richter.** (*Pharm. Zeit.*, 1912, 57, 125; through *Chem. Zentralbl.*, 1912, 1, 1148.)—The sulphite method given in the German Pharmacopœia for the estimation of formaldehyde was used. A solution of 10 grms. paraldehyde in 100 c.c. water is neutralised with $\frac{N}{1}$ potassium hydroxide solution, 2 drops of phenolphthaleïn solution being used as indicator, 20 c.c. of an aqueous solution containing 25 grms. crystallised sodium sulphite solution in 100 c.c. added, and $\frac{N}{1}$ hydrochloric acid to decoloration. From the quantity of hydrochloric acid used is subtracted the quantity required to decolorise a mixture of 100 c.c. water, 20 c.c. of the sulphite solution, and 2 drops of phenolphthalein solution. The difference must not exceed 9·1 (or 1·15, as the case may be) c.c. $\frac{N}{1}$ hydrochloric acid, corresponding to a maximum acetaldehyde content of 4 (or 0·5) per cent. acetaldehyde; 1 c.c. $\frac{N}{1}$ hydrochloric acid corresponds to 0·044 grm. acetaldehyde. Estimations of a number of commercial samples showed that paraldehyde with only a few tenths per cent. is easily obtainable. The author considers that paraldehyde of sp. gr. 0·998 to 1·00 may be required to show a solidification-point of 10° to 11° C., and not more than 0·3 per cent. acid, 0·5 per cent. acetaldehyde, and 0·1 to 0·2 per cent. metaldehyde, determined by evaporating 10 grms. of paraldehyde at 50° C. O. E. M.

Method of Distinguishing between Aldoses and Ketoses. M. Betti. (*Gazz. Chim. Ital.*, 1912, **42**, 288-294.)—The method is based upon the fact that β-naphthol-benzylamine forms crystalline compounds of amino-aldehydic character with aldoses, but does not react with ketoses. With *d*-mannose the reagent forms a compound, $C_{17}H_{13}ON : C_6H_{12}O_5$, which crystallises in white needles melting (with decomposition) at 207° to 208° C.; with *d*-galactose it yields small prisms melting at 206° C. (with decomposition); with *d*-glucose, white silky needles melting (with decomposition) at 192° C., and being more soluble in alcohol than the preceding compounds; and with rhamnose, white crystals melting (with decomposition) at 192° C. In using the reagent for the separation of dextrose and lævulose, a solution of 0·90 grm. of each of the sugars in dilute alcohol was added to a warm alcoholic solution of 2·5 grms. of the base, and the mixture left for twenty-four hours, and then allowed to evaporate spontaneously. The crystalline mass was triturated with a little water and filtered, and then washed on the filter. The filtrate and washings contained the lævulose. The residue, after being washed with petroleum spirit and crystallised from a large volume of alcohol, melted at 192° C., and by treatment with dilute hydrochloric acid yielded the dextrose again.

C. A. M.

Examination of Bituminous Materials. F. C. Garrett. (*J. Soc. Chem. Ind.*, 1912, 31, 314-317.)—The following methods of analysis were adopted: *Water* is determined by heating for five to six hours at 40° to 50° C., or by desiccation *in vacuo* over sulphuric acid for several days. The two methods give similar results, and a slight loss of organic matter takes place, but this is not important except in the case of pitches containing much water. *Sulphur* is determined by a modification of Eschka's method (ANALYST, 1906, 31, 52), the following results being obtained (the figures in brackets show the percentage amount of sulphur calculated on the organic matter only of the sample): Sulphur per cent. in petroleum, 1·39 (1·39); liquid pitch, 2·66 (2·66); soft pitch from Hatley's hole, Lot C, 2·88 (3·13); soft lake pitch, 3·62 (5·54); lake pitch, 3·81 (5·98); lake pitch dried by steam, 4·12 (6·52); land pitch, 3·55 (6·17); cheese pitch, 3·84 (6·38); iron pitch, 3·86 (6·27); impure pitch, seashore, 3·20 (7·67); manjak (Grahamite), 2·96 to 3·57 (3·13 to 3·74) in three samples; glance pitch, 5·12 (5·17); Barbados manjak, 0·86 (0·89); Cuban hard pitch (Grahamite), 8·47 (8·59); elaterite (elastic), 1·14 (1·16).

Proximate Analysis by Heating.—About 2 grms. of the sample are placed in a platinum crucible and heated with the lid on by the tip of a Bunsen-flame for half a minute after the disappearance of flames round the crucible lid. The time required is two and a half minutes, the bottom of the crucible being faintly red during the last half minute. The crucible is then heated to bright redness for two minutes in the flame of a blast Bunsen, cooled without removing the lid, weighed, and the loss of weight (less moisture) noted as "volatile hydrocarbons." The coke is next heated to redness till burnt off, the loss of weight being "fixed carbon" and the residue "ash." In the case of pitches which melt and froth over, smaller quantities are heated in a larger platinum crucible, and the first heating is spread over five minutes. The method allows of distinctions being made between, say, lake pitch and land pitch. The amount of volatile matter, divided by the fixed carbon, gives the "volatility factor," which in the lake pitches is above 5, and in the land pitches between 4 and 5. The following are typical of the results obtained :

PETROLEUM AND THE "PITCHES."

	Petroleum (Sp.Gr.,0·9226; Flash-point, 27° C.).	Liquid Pitch (Sp. Gr., 0·926).	Lake Pitch.	Land Pitch.	Iron Pitch.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Moisture	nil	nil	1·20	8·60	0·60
Volatile matter ...	97·94	93·00	53·30	47·70	50·60
Fixed carbon ...	2·01	7·00	10·25	9·85	11·20
Ash	0·05	0·02	35·25	33·85	37·60
Volatility factor ...	48·90	13·28	5·20	4·85	4·49

THE GRAHAMITES, MANJAK, ETC.

	Cuban Hard Pitch (Grahamite).	Trinidad Manjak (Grahamite).	Barbados Manjak.	Glance Pitch.	Elaterite (Elastic).
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Moisture	nil	0·80	0·50	nil	nil
Volatile matter ...	53·55	59·05	64·60	74·80	96·70
Fixed carbon ...	45·00	36·25	31·90	24·20	1·10
Ash	1·45	3·90	3·00	1·00	2·20
Volatility factor ...	1·19	1·62	2·00	3·09	87·90

Proximate Analysis by Extraction with Solvents.—A piece of filter-paper, 9 by 6 inches, is dried at 100° C. and weighed, folded lengthwise, about 2 grms. of the pitch, accurately weighed, placed on the central portion of the paper and spread in a very thin layer, the sides of the paper turned over, and the whole rolled from each end towards the centre. The cylinder thus prepared is tied with a weighed platinum wire and introduced into a Soxhlet tube, and extracted with acetone till the pitch, on two successive weighings, after draining and drying for two hours at 100° C., loses less than 10 mgrms. during twelve hours extraction. The loss is recorded as "petrolene," and the extraction then continued with chloroform, which similarly extracts the "asphaltene." The residue is ignited and the mineral matter weighed, the difference between 100, and the sum of the petrolene, asphaltene, and the mineral matter being the "organic matter not bitumen." This usually consists of plant remains. The following are some of the results obtained:

ANALYSES BY SOLVENTS.

		Liquid Pitch.	Lake Pitch.	Land Pitch.	Iron Pitch.	Manjak (Grahamite).	Barbados Manjak.	Glance Pitch.
		Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Moisture		—	—	—	0·67	—	—	—
Petrolene	bitumen	90·95	39·22	35·23	35·82	17·06	38·72	34·64
Asphaltene		5·10	16·69	10·42	17·03	80·70	56·27	64·84
Organic matter not bitumen		—	8·95	19·64	9·13	—	4·04	—
Ash		—	35·12	34·71	37·33	2·24	0·97	0·83

A. R. T.

Estimation of Cellulose in Woods and Textile Fibres. J. König and F. Hühn. (*Zeitsch. Farb. Ind.*, 1911-1912. Series of ten articles.)—The authors have reinvestigated the various analytical methods proposed or adopted for cellulose estimation, and give their preference to *acid* methods of digestion, especially for elimination of unsaturated and oxidisable groups (lignone). They have particularly studied the methods of König (digestion with glycerolin sulphuric acid at 137° C., followed by alkaline oxidation) and Tollens-Dmochowski (digestion of "crude fibre" with strong nitric acid). The weights obtained in either case show a considerable deficiency (10 to 12 per cent.) from those of the generally adopted methods. The discrepancy in the former case (König) is explained as due to removal of "hemihexosanes" from celluloses reputed as "normal." In the latter case Tollens "corrects" the figures obtained by multiplying by the factor 1.1, and the present authors see no ground of criticism of such a "correction."

These two methods are selected and commended, and the methods of ordinary practice are adversely criticised.

C. F. C.

NOTE.—Either process leads to a product of degradation of doubtful relationship to the original cellulose. The process of chlorination (following *alkaline* hydrolysis) depends upon specific constitutional (and quantitative) relationships, has been therefore generally adopted, and must be still commended.

Modification of the Sulphonation Test for Creosote. E. Bateman. (*U.S. Dept. Agricult., Forest Service*, Circular 191, December 9, 1911; through *J. Soc. Chem. Ind.*, 1912, **31**, 425.)—The following modification of the sulphonation test is claimed to give more accurate results in a shorter time than the old method: Ten c.c. of the fraction of creosote under examination are treated with four successive portions of 10 c.c. each of $\frac{N}{37}$ sulphuric acid in a Babcock's milk-bottle, which is shaken for two minutes after each addition. It is then kept for an hour at 98° to 100° C., being meanwhile thoroughly shaken every ten minutes, and is subsequently cooled, filled to the mark with ordinary sulphuric acid, and whirled for five minutes in a Babcock's separator, after which the amount of unsulphonated residue is read upon the graduated scale. The reading, multiplied by two, gives directly the percentage of unsulphonated oil by volume. The size of the steam-bath used to maintain a constant high temperature during the sulphonation is immaterial, provided it be large enough to contain completely the bottle beneath its cover.

Estimation of Formaldehyde. E. Rimini and T. Jona. (*Giorn. Farm. Chim.*, 1912, **61**, 49; through *Chem. Zentralbl.*, 1912, I., 1147.)—The usual methods are examined, especially that of Riegler (*Zeitsch. anal. Chem.*, 1901, **40**, 92), depending on the conversion of formaldehyde into formalazine by hydrazine:

$$2CH_2O + H_2N.NH_2 = C_2H_4N_2 + 2H_2O.$$

The excess of hydrazine is determined gasometrically in the sense of the equation:

$$\begin{gathered}15N_2H_4.H_2SO_4 + 10HIO_3 = 15N_2 + 3H_2O + 15H_2SO_4 + 10HI \\ 10HI + 2HIO_3 = 6H_2O + 12I.\end{gathered}$$

Formalazine is slowly attacked by iodic acid, and low values may result. Rimini has simplified the method by working in an alkaline solution, when the reduction does not go beyond the formation of iodide:

$$3N_2H_4.H_2SO_4 + 2KIO_3 + 6KOH = 3N_2 + 2KI + 3K_2SO_4 + 24H_2O.$$

Formalazine is stable in alkaline solution, and nitrogen from the excess of hydrazine only is measured. The method has been further simplified by the substitution of mercuric chloride for hydriodic acid:

$$N_2H_4.H_2SO_4 + 6KOH + 2HgCl_2 = K_2SO_4 + 4KCl + 2Hg + N_2 + 6H_2O.$$

The formalin found by the Riegler method is shown to decrease with the duration of reaction—from 39·00 per cent. after two minutes to 28·40 per cent. after thirty-nine. Tables showing the satisfactory agreement of results by the standard methods with those by the authors' method are given. The estimation is carried out as follows: The volume of nitrogen evolved from 20 c.c. of 1·1 per cent. hydrazine sulphate solution by mercuric chloride is first determined in a Lunge nitrometer; 20 c.c. of the solution are then added to 5 c.c. of the formaldehyde solution (about 1·1 per cent.) and the excess of nitrogen with mercuric chloride measured. The results of the examination of commercial disinfectants containing formaldehyde are given.

O. E. M.

Estimation of Formic Acid in the Presence of other Acids by Means of Potassium Permanganate. A. Fouchet. (*Bull. Soc. Chim.*, 1912, 11, 325-328.)—Small quantities of formic acid (1 to 5 mgrms.) may be estimated in the presence of large amounts of acetic, propionic, butyric acids, etc., by the following method: A solution of 0·05 grm. of the sample in a little water, is added to a mixture of 40 c.c. of crystalline sodium carbonate (50 grms. per litre) and 20 c.c. of potassium permanganate (5 grms. per litre), whilst simultaneously a blank test is made with water instead of the solution. In the case of very dilute solutions of formic acid, the permanganate solution should be diluted (1 : 10), and the other reagents also diluted. The two flasks are immersed for exactly three minutes in boiling water, after which 20 c.c. of dilute (1 : 1) sulphuric acid and 50 c.c. of ferrous ammonium sulphate solution (20 grms. of the salt and 30 grms. of sulphuric acid per litre) are introduced, and the excess of the iron salt estimated by titration with 0·5 per cent. permanganate solution. The difference between the results obtained with each flask gives the amount of oxygen corresponding to the oxidised formic acid, each c.c. of the permanganate solution being equivalent to 1·25 mgrms. of oxygen and 3·51 mgrms. of formic acid. The test analyses quoted in detail show that the method gives accurate results.

C. A. M.

Estimation of Glucose in Leather. J. G. Parker and J. R. Blockey. (*J. Soc. Chem. Ind.*, 1912, 31, 268-269.)—The authors show that in the estimation of glucose in leather, higher results are obtained when normal lead acetate is used for precipitating the tanning matters, etc., than when basic lead acetate is employed. For instance, in one sample 8·7 per cent. of glucose was found with normal lead acetate as against 7·8 per cent. with basic lead acetate. The discrepancy is due to the fact that certain cupric reducing matters—*e.g.*, gallic acid, are not completely precipitated in the presence of acid. The aqueous extract from leather contains some free organic acid, acetic or lactic; consequently the precipitation of the gallic acid would be incomplete when normal lead acetate is used to an extent depending on the free acidity; on the other hand, basic lead acetate would neutralise the free acid, giving a more complete precipitation of the gallic acid, and a lower but more accurate value for the glucose. Lower results are also obtained if the acidity of the aqueous extract be neutralised with potassium hydroxide, using phenolphthaleïn as an external indicator, care being taken not to allow the neutralised solution to stand long before removing the tannins with the lead solution. Previous neutralisation lowers the results for glucose not only with normal, but also with basic lead acetate. It has also been shown that the acidity of the solution affects the precipitation not only of the gallic acid, but also of certain of the tannins—viz., the group of catechol tannins. This is an additional argument against the use of normal lead acetate. J. F. B.

Notes on the Gum Resins. H. Ingle. (*J. Soc. Chem. Ind.*, 1912, 31, 272-273.)—On the question of the "weathering" of kauri and copal gums, the author is inclined to think that the "weathered" gum is probably quite as useful to the varnish-maker as the "bold" gum, and that the varnish prepared from it is less likely to deteriorate by further oxidation. The effect of "weathering" on the gums is shown by a decrease in the iodine value and an increase in the acid value.

Estimation of Kauri, Manila Copal, and Dammar, in a Mixture.—As the acid values of these three gums are so different (kauri = 50 to 70, manila = 107 to 156, and dammar herrol = 30 to 32), it was thought that a separation might be effected by means of sodium carbonate solution. Ten grms. of gum were finely ground, and treated with 300 to 400 c.c. of a 5 per cent. solution of sodium carbonate, boiled, filtered, and washed with the carbonate solution; the filtrate was acidified, and the precipitate collected on a tared filter-paper. The results showed: Kauri gum, 6·1 per cent.; manila, 92·9 per cent.; dammar, 2·5 per cent.

Estimation of Dammar.—The author describes a method based on solubility, which may be used in combination with the above method. The sample is ground to powder, and one or more grams are treated in a beaker or flask with a mixture of 3 parts of alcohol and 1 part of benzene; the residue is filtered off, washed with the same mixture, and then dissolved in carbon bisulphide, the filter being washed three times with this solvent, and the solution collected in a tared flask; the solvent is evaporated, and the extract dried and weighed. After the extraction with alcohol and benzene, kauri and manila gums leave no residue soluble in carbon bisulphide. Dammar herrol gives from 36 to 44 per cent. of matter insoluble in alcohol-benzene, but soluble in carbon bisulphide. If the mixture of gums is in lumps, some of the dammar may be picked out, and the actual solubility value of the particular sample employed may be determined. In a mixture of the three gums the dammar may be estimated by this carbon bisulphide method, and the kauri and manila by the acid value and matter soluble in sodium carbonate; very satisfactory results are obtainable.

SOLUBILITY TABLE OF GUM RESINS.

Solvent.	Kauri.	Manila.	Dammar.
Acetone	Insoluble	Partially soluble	Insoluble
Alcohol	Partially soluble	Soluble	Insoluble
Alcohol and American turpentine	Soluble	Soluble	Partially soluble
Benzene	Insoluble	Insoluble	Partially soluble
American turpentine	95·5 per cent. insoluble	104·0 per cent. insoluble*	—
Amyl acetate ...	5·3 per cent. insoluble	11·5 per cent. insoluble	26·7 per cent. insoluble
Ethyl acetate ...	Soluble	Nearly soluble	Insoluble
Petroleum ether ...	18·6 per cent. soluble	12·1 per cent. soluble	—

J. F. B.

* Result attributable to the oxidation of the solvent.

Separation of Liquids, the Boiling-Points of which lie close together, or which form Mixtures of Minimum Boiling-Point. A. Golodetz. (*Chem. Zeit.*, 1912, **36**, 273-274, 297-298, 302-304.)—Young (*J. Chem. Soc.*, 1902, **81**, 709) effected the dehydration of ethyl alcohol by taking advantage of the fact that ethyl alcohol, benzene (or hexane), and water formed a ternary mixture, which boiled constantly at a temperature lower than the boiling-points of the single constituents, or of the mixtures of minimum boiling-point which alcohol forms with benzene and with water separately. No similar ternary mixture of minimum boiling-point has since been described, but many separations, otherwise difficult or impossible, may be effected by adding a suitable third liquid to the mixture it is desired to resolve, and then distilling. For example, toluene and acetic acid form a mixture of minimum b.-pt. (104° C.), the mixture containing 30 per cent. of acetic acid. Since benzene also forms with acetic acid a mixture of minimum b.-pt. (80° C.), pure toluene remains as the residue in the still when the toluene-acetic acid mixture is distilled with a small excess of benzene. Since a mixture of toluene and water in the proportions 4 : 1 boils constantly at 84° C., the toluene-acetic acid mixture may also be resolved by distilling with water. In this case it is the acid which is separated in a pure condition, and if only a small excess of water be employed, as much as half the acid may be recovered of 99·9 per cent. strength. Similar methods serve for the separation of liquids which do not form mixtures of minimum b.-pt., but which boil at temperatures so nearly the same that separation by ordinary fractional distillation is impossible. For example, the last case may be reversed, and toluene made use of to prepare acetic acid of high strength from more dilute aqueous solutions. Another example of the resolution of mixtures of minimum b.-pt. is that of benzene and methyl alcohol by the agency of carbon bisulphide, which forms with methyl alcohol a mixture of still lower b.-pt.; whilst among mixtures of liquids with b.-pts. lying very close together, the most striking separation is that of amyl bromide (b.-pt. 118·5° to 119° C.) and ethyl butyrate (b.-pt. 119·9° C.) by distillation with propyl alcohol, which forms a mixture of minimum b.-pt. with amyl bromide.

G. C. J.

Ester Method for the Detection of Mono-Amino-Acids together with Polypeptides. E. Abderhalden and R. Hanslian. (*Zeitsch. physiol. Chem.*, 1912, **77**, 283-288.)—The ester method of separating amino-acids resulting from the cleavage of polypeptides, etc., the esters being formed by saturating the alcoholic solution of the acids with dry hydrochloric acid gas, yields trustworthy results, and there is no risk of producing further quantities of amino-acids during the esterification. It is usual to cool the mixture during the latter operation, but experiments with polypeptides of high molecular weight showed that further decomposition does not take place when the mixture is not cooled. Silk fibroin yields no soluble nitrogenous product when esterified, but when the alcoholic solution is saturated with hydrochloric acid and boiled for six hours, considerable quantities of amino-compounds are formed. In the case of casein, boiling the esterified mixture results in only a very small increase of the amino-compounds.

W. P. S.

Estimation of Mercury in Smokeless Powder. J. D. Berkhout. (*Zeitsch. ges. Schiess- u. Sprengstoffwesen*, 1912, 7, 6[illegible].)—In a 50 c.c. wide-mouthed stoppered bottle 2·5 grms. nitrocellulose or nitroglycerine powder are covered with 20 c.c. acetone and kneaded with a glass rod. The sample is sufficiently softened in eighteen hours, and is then transferred to a layer of fine copper oxide in a mortar; the bottle is rinsed out with copper oxide into the mortar. The powder and copper oxide are mixed with a spatula until they form a fine-grained dry mass, most of the acetone having evaporated; this is transferred to a soft-glass tube of 50 cm. length and 14 mm. bore, closed at one end. Ignited asbestos fibre, rinsings of copper oxide from the mortar, asbestos, 15 cm. of coarse copper oxide, and asbestos, are successively introduced, and the tube drawn out into five constrictions, 2 mm. in diameter and 5 cm. long, and four bulbs. The tube is closed with a rubber stopper and bent tube dipping into water. The tube and one bulb are encased in asbestos-lined sheet-iron; the three outer bulbs, projecting through an asbestos card, are water cooled. Heat is applied first to the coarse copper oxide, and gradually extended to the rest of the tube at such a rate that not more than two bubbles per second are given off. When gas evolution has nearly ceased, the capillary is sealed off between the tube and the first bulb, filled up with concentrated hydrochloric alcohol and alcohol, heated in the water-bath, and inverted upon a watchglass containing water. The globule of mercury is measured under the microscope or weighed. O. E. M.

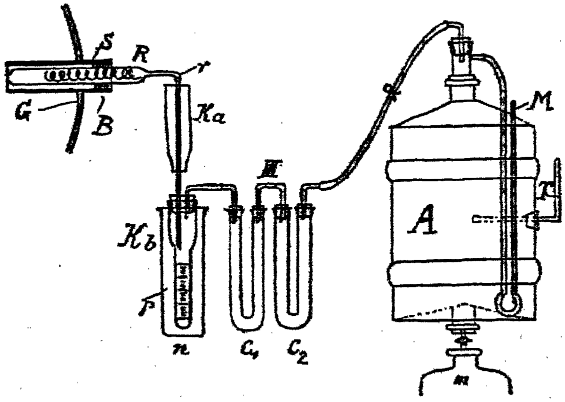
Estimation of Sulphur in Nitrocellulose. C. Kullgren. (*Zeitsch. ges. Schiess- u. Sprengstoffwesen*, 1912, 7, 89.)—The sulphur is present, as a sulphuric acid ester, in combination with nitrocellulose. After several of the known methods for the estimation of sulphur in nitrocellulose had been investigated and rejected, the sample was denitrated by heating it with 10 c.c. of concentrated hydrochloric acid for thirty minutes on the water bath, dried in a large porcelain boat, and burnt in a current of oxygen. The sulphuric acid was arrested by sodium carbonate on granulated quartz. A small quantity of sodium peroxide was added to the solution before the precipitation with barium chloride, to insure that all sulphur is present as sulphuric acid. Nitrocellulose boiled for one-quarter of the usual time showed 27·8 mgrms. of barium sulphate per grm. of substance, unboiled about the same, boiled for one-half the usual time, 17·2, and completely boiled, 14·4. O. E. M.

Influence of Lucidol (Benzoyl Peroxide) on Certain Qualitative Tests for Oils. Atz. (*Chem. Rev. Fett- u. Harz. Ind.*, 1912, 19, 70-72.)—If excessive quantities of the new bleaching agent, "lucidol," have been applied to olive or cottonseed oils, colorations may be obtained with Baudouin's or Soltsien's reagents, which have some resemblance to those given by sesamé oil. On the other hand, in certain tests for other oils—*e.g.*, Becchi's reaction for cottonseed oil—the coloration is produced with the same intensity in the presence of "lucidol," but the occurrence of the reaction is retarded. In the case of some other colour reactions, the intensity of the coloration is sometimes reduced, as, for instance, in the reactions for cottonseed and sesamé oils. It is, therefore, necessary to take the possibility of the presence of "lucidol" into account in testing oils, such as olive oil, for the presence of small quantities of other oils. C. A. M.

Estimation of Petroleum Spirit and Benzene Hydrocarbons in Turpentine Oil. J. Marcusson. (*Chem. Zeit.*, 1912, **36**, 413-414; 421-422.)—In Burton's method of estimating petroleum spirit in turpentine oil the sample is treated under specified conditions with fuming nitric acid, in which turpentine oil is completely soluble, whereas in the presence of petroleum spirit one portion of the latter (naphthenes and paraffin hydrocarbons) is undissolved, whilst another part (aromatic hydrocarbons) is dissolved, but may be separated from the nitro-derivatives of the turpentine oil by means of its insolubility in water. Criticisms have been brought against this method by Herzfeld (*Chem. Zeit.*, 1910, **34**, 885) and others, but the author shows that it gives good results, which are independent of the nature of the petroleum spirit, which may contain but little benzene hydrocarbons (as in the case of Russian and Pennsylvanian products) or be rich in those hydrocarbons (Indian and Galician petroleum spirits). The method is, therefore, to be preferred to that of Eibner and Hue (ANALYST, 1910, **35**, 358), which, as its originators showed, is not applicable to the heavy Indian petroleum spirits, such as Sanga oil, which at the present time are extensively used in the preparation of substitutes for turpentine oil. In testing a mixture 10 c.c. of the sample are introduced, little by little, during the course of thirty minutes, into 30 c.c. of fuming nitric acid (sp. gr., 1·52) chilled to − 10° C. The mixture is left for fifteen minutes, and then treated with 75 to 80 c.c. of concentrated nitric acid (sp. gr., 1·4); and should no oily layer appear upon the surface of the liquid, the absence of petroleum spirit is indicated. For the detection of benzene hydrocarbons, the solution is poured into 150 c.c. of water in a 500 c.c. flask with a neck graduated in $\frac{1}{10}$ths c.c., and heated for fifteen minutes on the water-bath, after which the flask is allowed to stand for some hours. The separation of heavy reddish-brown oily drops (not merely slight resinous masses) denotes the presence of benzene hydrocarbons. For an approximate estimation of these hydrocarbons, the product of the reaction is treated with sulphuric acid (sp. gr., 1·6) until the oily nitro-derivatives are driven into the graduated neck of the flask, where their volume may be read. In test estimations with mixtures containing from 10 to 50 per cent. of benzene, toluene, or xylene, the results agreed within 4·5 per cent. of the theoretical amount, whilst with technical benzene preparations ("solvent naphthas") the maximum difference was 3 per cent. If the presence of more than 50 per cent. of benzene hydrocarbons be suspected from the bromine value, etc., the heating of the diluted nitric acid solution should be omitted, since, in the absence of sufficient turpentine oil, the hydrocarbons in these solvent naphthas are attacked under these conditions. On the other hand, omission of the heating causes the proportion of nitro-derivatives to be too high, and to correct this the result should be divided by the factor 1·15. The figures will then correspond within about 4 per cent. with the theoretical amounts. C. A. M.

Application of the Formolite Reaction to the Analysis of Paraffin Wax. F. Sommer. (*Petroleum*, 1912, 7, 409; through *Chem. Zentralbl.*, 1912, I., 1151.)—The oils contained in commercial paraffin wax determine its tendency to turn yellow and its transparency, both important factors in a valuation. Drainings from the paraffin sweating process were repeatedly sweated, cooled to below 0° C., and pressed in filter-cloths. The resulting oil contained only small quantities of saturated hydrocarbons; its iodine value was 444; three-quarters of it dissolved in concentrated sulphuric acid, and it gave 63 per cent. of formolite. To represent the transparency in figures, the liquid hydrocarbons must be determined; the yellowing tendency is, however, a function of the formolite number, as the table of results shows. This is determined as follows: To 20 grms. of the molten paraffin in 20 c.c. of concentrated sulphuric acid are gradually added 20 c.c. of formaldehyde. The dark red mixture is heated on the water-bath for twenty minutes, poured into a porcelain dish, left on the water-bath until paraffin and reaction-mixture separate, and cooled; the cake of paraffin is removed, and the liquid beneath diluted and shaken out with chloroform. The chloroform extract is evaporated, the residue dried at 105° C., and the formolite weighed. All samples examined were exposed to diffused daylight. The formolite number is suggested as a criterion of the quality of paraffins; factories having a uniform supply of paraffin oil can compare colorimetrically the chloroform solution with a normal colour. The authors state that a paraffin wax with more than 0·75 per cent. oil is always opaque. O. E. M.

Estimation of Tar in Producer Gas. A. Gwiggner. (*Chem. Zeit.*, 1912, **36**, 461-462.)—The apparatus of Tieftrunk is useless for the estimation of tar in hot-producer gas. The apparatus now figured serves well for this purpose. A wrought iron tube, *S*, is screwed into the gas main, *G*, to support the tar-filter, *R*, which is formed from combustion tube 30 to 35 mm. in diameter, packed with slag wool, and drawn out at one end and constricted at the other. Its wide portion is about 40 cm. long. The condenser tube, *r*, carries a removable condenser, *Ka*, and leads into the measuring tube, *P*. *Ka* and *Kb*, the beaker in which *P* stands, are filled with ice water. C_1 and C_2 are tubes containing calcium chloride, and *A* is an aspirator of at least 10 litre content. Before use *R* is weighed separately; *r* and *P*, disconnected from the condenser, *Ka*, are weighed together; and C_1 and C_2 are weighed separately. Even with a very brisk stream of gas, no visible fog reached C_1, and the increase in the weight of this tube is due to moisture alone. The increase in weight of the system $r+P$ is due to light oil and moisture, and the latter can be separately estimated by washing down *r* with benzene or light petroleum, diluting the contents of *P* somewhat with the same solvent, and reading off the water by means of the graduations on *P*. The increase in the weight of *R* is due to soot, tar, and some moisture. To determine these separately, *R* is



connected to apparatus similar to the system $r+P+C_1$, and a current of hot dry air is led through the whole train of apparatus. The moisture finds its way to the graduated tube and calcium chloride tube. Usually a little light oil finds its way into the graduated tube, and is separated from the water in the manner already described. The tube R is next weighed, its contents washed with benzene so long as the escaping benzene is discoloured, and is finally dried and weighed. The loss of weight on treatment with benzene gives the content of tar, and the increase of weight as compared with the weight at the commencement of the test gives the content of soot. Some ten weighings in all are necessary, but it is said that no simpler method will give results of any value where the gas to be sampled is producer gas at temperatures in the neighbourhood of 600° C. G. C. J.

New Uric Acid Reaction. D. Vitali. (*Boll. Chim. Farm.*, 1911, **50**, 799; through *Chem. Zentralbl.*, 1912, I., 1252.)—Ganassini (*Boll. Chim. Farm.*, 1908, **47**, 715) has stated that uric acid in alcoholic solution gives, with various oxidising agents an oxidation product which forms a characteristic blue basic salt with zinc salts. The author finds that the oxidation may be effected with sodium peroxide, and that as little as $\frac{1}{10}$ c.c. of freshly prepared alkaline uric acid solution then gives a positive reaction. A reaction is not obtained with other oxidising agents, such as hydrogen peroxide, turpentine, turpentine + blood, pus, and copper sulphate + hydrocyanic acid. Permanganate solution produces a yellow coloration, and, on the addition of zinc sulphate, a white precipitate insoluble on warming. If copper sulphate is substituted for zinc sulphate, a green precipitate, or with a larger quantity, a blue precipitate, is formed; cobalt chloride, and a nickel salt give respectively blue-violet and yellow-green precipitates, reactions which are very sensitive, and can be used to distinguish between nickel and cobalt salts. The precipitate with neutral antimony acetate is brown-red; with mercuric chloride white, or on warming, vermilion; with silver nitrate metallic silver is obtained. The blue colour of the basic zinc urate is destroyed by the addition of sulphur or of hydrocyanic acid. The reducing action of zinc or of sodium hydrogen sulphite in the alkaline reaction mixture does not cause decoloration. O. E. M.

INORGANIC ANALYSIS.

Volumetric Estimation of Beryllium. B. Bleyer and A. Moormann. (*Zeitsch. anal. Chem.*, 1912, **50**, 360-367.)—Normal beryllium salts are so far hydrolysed in aqueous solution that their solutions have a fairly strong acid reaction, and will set free iodine from a solution of potassium iodide and iodate. If sufficient of this solution be added, the hydrolysis is quantitative and the beryllium may be estimated from the iodine set free—$BeCl_2 = I_2$. If the iodine be distilled over into potassium iodide solution, using a curent of hydrogen to drive the last of the iodine vapour out of the apparatus, the results, as determined by titration of the iodine with thiosulphate, will be constantly 1 per cent. below those indicated by theory. Omission of the current of hydrogen doubles the error, which in such circumstances is also less constant.

In pure aqueous solution, salts of beryllium may be estimated by titration with standard caustic alkali, using phenolpthaleïn as indicator. Beryllium sulphate must be converted into chloride by addition of barium chloride before it can be titrated in this way. In the ordinary course of analysis the beryllium salt most often presents itself in solutions containing excess of acid. In such cases concentrated caustic soda, free from carbonate, is added to the solution drop by drop until the precipitate of beryllium hydroxide first formed has re-dissolved as beryllate, and the alkaline solution is made up to known bulk. Aliquot portions are then titrated with standard acid, using phenolpthaleïn and methyl orange as indicators. The difference between the two titrations is the measure of the beryllium—Be = 2HCl. The solution should not contain much more than 0·03 per cent. of beryllium, as otherwise the end point of the phenolphthaleïn titration is a little difficult to determine. The titration should be made at a temperature of 30° to 40° C., and it has to be remembered that towards the end of the methyl orange titration the reaction proceeds somewhat slowly. The test numbers which accompany the paper are all almost exactly 1 per cent. low.

G. C. J.

Estimation of Boric Acid in Nickel Baths. A. Wogrinz and J. Kittel. (*Chem. Zeit.*, 1912, **36**, 433-434.)—Experiments are described which show that the amount of boric acid in nickel-plating baths cannot be estimated with any approach to accuracy by the method of Rose, or by that of Marignac. Methods depending on the titration of the boric acid in presence of a large quantity of some polyhydric alcohol give sufficiently accurate results, but only after removal of the ammonium salts, the concentration of which in nickel-baths may be considerable. The sample (100 c.c.) is first electrolysed to throw out nickel and decompose citric acid, which is nearly always present in such baths. The solution is then made strongly alkaline by addition of 10 grms. of caustic soda and evaporated in a nickel dish to decompose ammonium salts. The residue is made acid with hydrochloric acid and the solution diluted to 25 or 30 c.c., and heated to expel carbon dioxide. After allowing to cool, the solution is exactly neutralised to methyl orange, 100 c.c. of a 10 per cent. solution of mannitol is added, and the boric acid titrated in the usual manner with $\frac{N}{5}$ caustic alkali, using a considerable quantity of phenolphthaleïn as indicator. The results are from 0·5 to 1 per cent. high, calculated on the boric acid. G. C. J.

Sources of Error and the Electrolytic Standardisation of the Conditions of the Iodide Method of Copper Analysis. A. W. Peters. (*J. Amer. Chem. Soc.*, 1912, **34**, 422-454.)—The various methods for the standardisation of sodium thiosulphate may be classified by groups, and the differences between the values found by the different groups far exceed the limits of error of the methods of the same group. Thus, for the estimation of copper, the thiosulphate must be standardised against a copper solution, and accurate results cannot be expected by employing a factor based on an iodine standard. The author has worked out a rapid electrolytic method for standardising a copper solution for this purpose. An alkaline electrolyte is employed, the reagent containing 35 grms. of Rochelle salt and 25 grms. of purified potassium hydroxide per 100 c.c.; 10 c.c. of the copper solution are mixed with 10 to 20 c.c. of this reagent, and a quantity of a saturated solution of potassium cyanide, ranging from 0·4 to 1·0 c.c. is added. The cyanide controls the rate of deposition and the hardness of the deposit, but if too large a quantity be employed, the time required for complete precipitation is lengthened. Rapid deposition is obtained by using a high voltage, high current density, and keeping the temperature near the boiling-point. For preparing solutions of copper for titration by the iodide method, the copper is heated in an Erlenmeyer flask with the smallest necessary quantity of slightly diluted nitric acid. The solution is diluted, powdered talc is added, and the mixture is boiled vigorously for five to ten minutes. The rapid ebullition quickly removes nitrous acid, and the liquid is then cooled and diluted. Sulphuric acid may be added to the nitric acid solution when a solution of the sulphate is desired. The accuracy of the results obtained by the iodide method is influenced by varying concentrations of mineral acids, and by the presence of large amounts of salts. In the latter case, as, for instance, when the alkaline copper solutions from sugar analyses have to be titrated, sulphuric acid must be used instead of acetic acid in order to obtain good results. For instance, when the total volume of reduction-liquor was 60 c.c., consisting of 20 c.c. of Ahlin's alkaline tartrate solution, 20 c.c. of Fehling's copper sulphate, and 20 c.c. of water, the precipitated cuprous oxide could be filtered off and washed, giving a total volume of 70 to 75 c.c. of filtrate. The addition of 3·45 to 5·0 c.c. (best 4 c.c.) of concentrated sulphuric acid to this volume of liquid gave good results on titration, but smaller amounts were unsatisfactory. In working with ordinary solutions of copper (*i.e.* where no alkali has to be neutralised), normal results are obtained with 1 c.c. of concentrated sulphuric acid in 60 of the final liquid, or with 1 c.c. of strong acetic acid in 6 of the solution. A further essential factor for success is the presence of a sufficiently large quantity of potassium iodide; this is best employed in the form of a saturated solution containing about 1·1 grm. of the salt per 1 c.c. In titrations completed at a total volume of 60 c.c., 5 c.c. of the iodide solution are required; for titrations at a final volume of 120 c.c., 6 to 10 c.c. of iodide are used. Lastly, the question of the end-point requires careful attention, since it is not readily observed in presence of the cuprous iodide. So long as a drop of thiosulphate falling into the solution produces a white area, the end-point is not reached. It is best to have a burette containing standard iodine always at hand, and to verify the end-point by titrating back again immediately. Provided the above precautions be taken, the results of the iodide method are quite unaffected by the concentration of the copper. J. F. B.

Relation of the Refractive Index of Soda Lime Glasses to their Chemical Composition. E. W. Tillotson, Jun. (*J. Ind. Eng. Chem.*, 1912, **4**, 246-249.)—A number of batches of glass of known chemical composition were prepared, and the density and refractive index of each was determined, and the specific refractivities calculated by the formula of Lorenz and Lorentz (*Wied. Ann.*, 1880, **9**, 641; 1882, **11**, 70) $\frac{n^2-1}{n^2+2} \times \frac{1}{d} = K$. On plotting the values of K thus obtained as abscissæ in a system in which the ordinates represented the chemical composition of the glasses, which ranged from $Na_2O.3\ SiO_2$ to $CaO.SiO_2$, K was found to be a linear function of the chemical composition, at any rate until the molecular ratio of lime to soda reaches 3 : 2. At this point there is a slight change in the direction of the curve, but in very few glasses does the ratio of lime to soda exceed this limit. Since the specific refractivity is linear, it may be computed by means of the following equation $K = \frac{P_1K_1}{100} + \frac{P_2K_2}{100} + \frac{P_3K_3}{100}$, etc., in which P_1, P_2, P_3, etc., are the percentages of the oxides and, K_1, K_2, K_3, etc., the empirically determined specific refractivities of the oxides. For soda lime glasses these are as follows:

SiO_2 . . . 0.1220, CaO . . . 0.1210, Na_2O . . . 0.1302.

G. C. J.

Use of the Mercury Cathode, particularly in the Electro-Analytical Separation of Metals. P. Baumann. (*Zeitsch. anorg. Chem.*, 1912, **74**, 315-350.) The author has applied his apparatus (ANALYST, 1911, **36**, 529) to the separation of mercury from bismuth, mercury from copper, and silver from bismuth, all of which separations may be made rapidly at ordinary temperatures. For the reasonably rapid separation of copper from antimony, copper from cadmium, and bismuth from cadmium, a temperature of about 70° C. is necessary. It is probable that cadmium could be separated from zinc by means of the mercury cathode; but the separation is so slow, and requires so much of the analyst's attention, that the method could serve no useful purpose. One difficulty in the use of a mercury cathode is the need of considerable quantities of ether which shall be without action on mercury. Simple distillation of the ether does not get over the difficulty, as, within a few days, it re-acquires the faculty of giving rise to a black film on the surface of mercury with which it is brought in contact. If the ether be stored in a large distilling flask over caustic potash, and distilled in small quantities as wanted, the trouble is overcome, and the distillate remains for three or four weeks without action on mercury.

G. C. J.

Perchloric Acid in Electrochemical Analysis. W. S. Hendrixson. (*J. Amer. Chem. Soc.*, 1912, **34**, 389-392.)—In a recent paper by H. Golblum (*Zeitsch. anal. Chem.*, 1911, **50**, 741) on the electrolytic decomposition of nickel and cobalt perchlorates, it was shown that perchloric acid is remarkably stable, and therefore suitable for general purposes in electrolytic work. The only reducing agents which decompose perchloric acid in solution appear to be freshly precipitated ferrous oxide, sodium thiosulphate, and, to a small extent, some oxides of nitrogen; in fact, in aqueous solutions perchloric acid exhibits only the properties due to its primary ions, and there are no side reactions due to the decomposition of its negative ion. Besides being stable, it is also a "strong" acid—*i.e.*, highly dissociated, and metals may easily be converted into their perchlorates for electrolysis. Perchloric acid readily dissolves many oxides of the heavy metals, and in other cases the nitrates may be converted almost quantitatively into perchlorates by evaporation with an excess of perchloric acid. From potassium nitrate 94 per cent. of the nitric acid was expelled, and from copper nitrate 98 per cent. The author describes the electrolytic deposition of copper, silver, and cadmium from perchloric acid solutions. Pure copper oxide was dissolved in a small excess of perchloric acid, and subjected to electrolysis in a platinum dish with a rotating anode; the results were perfectly satisfactory. Silver was dissolved in nitric acid, and the resulting silver nitrate evaporated twice on the water-bath with water and perchloric acid, a trace only of nitric acid being left in the perchlorate. In the subsequent electrolysis silver oxide tended to collect on the anode, but was redissolved, especially on warming, long before the current was stopped. With cadmium also a satisfactory deposit was obtained from a solution of the oxide in perchloric acid. The acidity in all these experiments ranged from 0·03 to 0·4 normal, mostly round about 0·1 normal. J. F. B.

Standardisation of Potassium Permanganate Solution by Sodium Oxalate. R. S. McBride. (*J. Amer. Chem. Soc.*, 1912, **34**, 393-416.)—On behalf of the Bureau of Standards, the author has subjected the process of standardising permanganate solution by means of sodium oxalate to a complete revision, studying the effect of every conceivable variation and source of error separately—viz., temperature, acidity, volume of solution, rate of addition of the permanganate, access of air, and presence of added manganous sulphate. In connection with the question of the excess of permanganate present when the end-point is determined, the author has confirmed the accuracy of Bray's method, according to which the titrated solution is cooled, treated with potassium iodide, and the iodine liberated is titrated back with 0·02 N thiosulphate. He found that the residual oxidising power of the small excess of permanganate required to establish an end-point remains unchanged for at least half an hour, and consequently the lapse of five to ten minutes required for cooling the solution is quite negligible. Using this control, the author found that if the end-point be approached slowly in a solution at a temperature above 60° C., the depth of colour is proportional to the excess of permanganate present. The chief possible source of error is the continued existence of manganese in the higher stages of oxidation, resulting in an apparent loss of oxygen, due to low temperature, large bulk of the solution, too rapid addition of permanganate (especially at the start or just before the end-point), and insufficient stirring. On the other hand, the addition of manganous sulphate decreases the consumption of permanganate. The author finally recommends the following procedure: In a 400 c.c. beaker dissolve 0·25 to 0·3 grm. of sodium oxalate in 200 to 250 c.c. of hot water (80° to 90° C.), and add 10 c.c. of dilute (1 : 1) sulphuric acid. Titrate at once with $\frac{N}{10}$ potassium permanganate solution, being very careful to stir the liquid vigorously and continuously. The permanganate must not be added more rapidly than at the rate of 10 to 15 c.c. per minute, and the last 1 to 0·5 c.c. must be added drop-wise, with particular care to allow each drop to be fully decolorised before the next is introduced. The excess of permanganate used to cause an end-point colour must be estimated by matching the colour in another beaker containing the same bulk of acid and hot water. The solution should not be below 60° C. by the time the end-point is reached; more rapid cooling may be prevented by standing the beaker on a hot plate during titration. The use of a small thermometer as a stirring-rod is recommended. The author employed a weight burette delivering a drop of 0·03 c.c. for his titrations; the influence of temperature changes due to the steam rising from the hot liquid is thereby avoided.

The accuracy of the above method is claimed to be within 0·1 per cent.; probably the error does not exceed 0·05 per cent. J. F. B.

Direct Determination of Small Amounts of Platinum in Ores and Bullion. F. P. Dewey. (*J. Ind. Eng. Chem.*, 1912, 4, 257-258.)—The old method of parting by means of sulphuric acid, re-alloying with silver, and parting with nitric acid, is quite useless for the determination of small amounts of platinum. The second weighing may possibly exceed the first, even when traces of platinum are present, and other members of the platinum group may go into solution in nitric acid and be estimated as platinum. Small amounts of platinum (and gold) may be estimated accurately and conveniently as follows: The silver alloy first obtained in the regular course of assay is parted with nitric acid in the usual way, platinum, if present in small amount, passing into solution with the silver. The solution is evaporated, if strongly acid, and then diluted, and about 1 or 2 c.c. of strong hydrogen sulphide solution diluted to 15 to 30 c.c. is added. After three or four hours, or, better, after standing overnight, the precipitate of silver sulphide containing all the platinum metals, if these were present in small amount, is filtered off, dried, and ignited. The resulting metallic sponge is wrapped in lead-foil, cupelled, and parted by means of strong sulphuric acid. If the amount of fine metal is considerable, the actual platinum may be determined by the double chloride method. In the examination of fine silver for traces of platinum and gold, 100 grms. or more of the silver is dissolved in nitric acid, the bulk of the acid evaporated, and the gold and platinum, with some silver, precipitated in the diluted solution (250 c.c.) by 5 c.c. of strong hydrogen sulphide diluted to 50 c.c. The sulphide precipitate is filtered off, ignited, and cupelled, the lead parted with nitric acid, and the gold weighed. The platinum in solution is then precipitated with hydrogen sulphide in the manner already described, and parted from silver by means of sulphuric acid. In this way two purchases of fine silver were found to contain, in each 100 grms., 0·23 and 0·10 mgrm. of gold, and 0·55 and 0·14 mgrm. of platinum, respectively.

In the case of material containing a considerable amount of platinum, the well-known fact that platinum alloyed with silver is not entirely soluble in nitric acid must be considered. In such a case the gold must be realloyed with silver, and reparted two or three times.

The possibility of the application of the method to the estimation of noble metals in copper and similar materials is indicated, but no experiments in this direction are described. G. C. J.

Estimation of Gold, Silver, and Platinum. Trenkner. (*Metallurgie*, 1912, 9, 103.)—The assay of platinum by cupellation with lead requires an excessive temperature, and the bead formed contains lead and is brittle. Gold and platinum may be estimated to 0·05 per cent. by the following method. For the preliminary assay 500 parts by weight (500 mgrms. = 1,000 parts by weight for alloys with more than 25 per cent. gold + platinum; 1,000 mgrms. = 1,000 parts by weight for poorer alloys) are cupelled at 900° to 950° C. with 3 to 15 grms. lead, according to the amount of base metal present; from the more or less grey, flat, and rough appearance of the bead, the content of gold and platinum is judged, or is roughly determined by weighing the residue after dissolving in concentrated sulphuric acid. For the assay itself 500 parts by weight, with ten times as much silver (*cf.* Sharwood, *J. Soc. Chem. Ind.*, 1904, **23**, 412) as the platinum and gold found by the preliminary determination, are cupelled with lead; the bead, less the silver added, gives the weight of gold, platinum, and silver. The amount of silver lost is found by cupelling a known similar alloy. The bead is dissolved in 25 to 30 minutes in 25 c.c. of sulphuric acid of sp. gr. 1·84, which is heated, but not boiled; decanted and washed three times with water; the residue gently ignited and dissolved in a minimum quantity of *aqua regia*, most of which is then driven off; the liquid is diluted with water, and the silver chloride removed by filtration; 15 c.c. of hydrochloric acid of sp. gr. 1·19 are added to the filtrate, the volume of which is about 150 c.c., and the gold precipitated with 1 grm. of hydrazine hydrochloride, allowed to stand with occasional stirring for one hour, filtered, ignited, and weighed. To remove any platinum it is fused on a cupel with a small quantity of lead and five to eight times its weight of silver, and the platinum and silver dissolved out by boiling with nitric acid. The platinum is precipitated from the gold filtrate by ammonia (Jannasch, *Ber.* 1903, **37**, 1980) or with potassium hydroxide. O. E. M.

Estimation of Sulphates in Solution by the Physico-Chemical Volumetric Method. A. Bruno and P. T. d'Auzay. (*Comptes rend.*, 1912, **154**, 984-986.)—Dutoit and Duboux (ANALYST, 1908, **33**, 90) proposed a method for the estimation of sulphates, notably in wines, by adding to the liquid $\frac{N}{4}$ baryta water, and measuring the electrical conductivity after each addition, plotting the values in the form of a curve. Theoretically, such a curve should show a minimum point corresponding to the total precipitation of the sulphates. On applying this method to a number of French wines, however, the authors found that the results were inaccurate in about three-quarters of the cases. They have therefore tested the method on aqueous solutions containing sulphuric acid, potassium sulphate, potassium bitartrate, tartaric acid, and malic acid, alone or in the form of mixtures.

The results in the case of solutions containing sulphates were classified in the following manner: 1. The curve shows no minimum point. This is the case with a pure solution of neutral potassium sulphate, where the end of the precipitation is shown by the intersection of two ascending straight lines. 2. The curve shows a minimum point. This is the case with all the other solutions in which sulphates were present, and the results are subdivided as follows: (1) The minimum corresponds actually and exclusively to the total precipitation of the barium sulphate with (*a*) sulphuric acid alone; (*b*) sulphuric acid in presence of potassium bitartrate, in which case the curve consists of three straight lines, one of them descending. (2) The minimum point never corresponds to the end of the precipitation with (*c*) potassium bisulphate alone; (*d*) sulphuric acid in presence of tartaric or malic acid. In the case of (*c*) the minimum point coincides with the precipitation of half of the total sulphuric acid. In the case of (*d*) the minimum point always occurs after the total precipitation of the sulphate, and the neutralisation of the organic acid only takes place after that of the sulphuric acid. (3) Lastly, the minimum point may coincide with the total precipitation in the case of potassium sulphate, neutral or acid, in presence of bitartrate or organic acids; but the phenomenon is quite fortuitous and unreliable, depending on complex equilibria which may become established. This case corresponds to wines. Similar conclusions were arrived at in the case of dilute alcoholic solutions, and the proposed method, therefore, cannot be used for the estimation of sulphates in wines. J. F. B.

Modification of the Method of Arnold and Hardy for the Rapid Estimation of Sulphur in Siderurgical Products. F. Giolitti and M. Marantonio. (*Rassegna Mineraria, Metallurgica e Chimica*, 1911, 35; through *Chem. Zentralbl.*, 1912, 1, 1250.)—The method of Arnold and Hardy (*Chem. News*, 1888, **58**, 41) consists in converting the sulphur into sulphuretted hydrogen, and observing how far the precipitation of lead sulphide progresses in a series of vessels containing lead acetate solution of known strength. The authors suggest (1) an improved form of vessel, more easily cleaned than the original one; and (2) the passage of the gas through a heated tube, to decompose organic sulphur compounds incapable of reaction with lead acetate, before it reaches the vessels. Sample analyses, with controls in which the sulphur is determined independently, show the utility of the method.

O. E. M.

Estimation of Sulphur in Insoluble Sulphides. T. S. Warunis. (*Ber.*, 1912, **45**, 869-870.)—The method previously described for the estimation of sulphur in coal (ANALYST, 1911, **36**, 375) has been extended, with slight modification, to the estimation of sulphur in insoluble sulphides. The substance (0·5 grm.) is heated in a porcelain crucible with a mixture of 4 parts sodium carbonate and 3 parts copper oxide, at first at a low heat, but finally with the full flame of a Bunsen burner. The mixture is frequently stirred during the heating, which should be continued for two hours. The contents of the crucible, when cool, are boiled out successively with water, sodium carbonate solution, and again with water, and the solution is filtered and acidified with hydrochloric acid. In absence of metals which form insoluble sulphates the contents of the crucible may be dissolved at once in hydrochloric acid. After separation of silica, if present, by evaporation, the hydrochloric acid solution of the sulphate is precipitated hot with barium chloride, and the precipitate treated in the usual manner. An analysis of pyrites gave 53·16 per cent. sulphur against 53·04 by Fresenius's method; and an analysis of mercuric sulphide gave 13·75 per cent. sulphur, as compared with 13·65 per cent. obtained after oxidation by means of potassium chlorate and concentrated nitric acid. G. C. J.

Rapid Determination of Vanadium in Steel. F. Garratt. (*J. Ind. Eng. Chem.*, 1912, **4**, 256-257.)—The following modification of Johnson's method ("Chemical Analysis of Special Steels," p. 8) is found by the author to be preferable to any other for routine work. The steel (2 grms.) is dissolved in 50 c.c. of dilute sulphuric acid, and 5 c.c. of nitric acid is then added to oxidise the iron and any tungsten which may be present. The solution is boiled until nitrous fumes are no longer evolved, or, in the case of tungsten steels, until the separated tungstic acid is of a pure yellow colour. The solution is next diluted to about 150 c.c., filtered from tungstic acid, if this is present, and cooled to room temperature. It is then further diluted to about 350 c.c., ferrous sulphate is added, and then dilute permanganate from a burette until a permanent pink tint is just reached. Potassium ferricyanide is added as indicator, and the vanadium titrated with standard ferrous ammonium sulphate. Blanks must be run on steels of similar composition, and consistent end-points adhered to in titrations. The author agrees that for umpire work, and in any case where a sample concerning which nothing is known has to be analysed, a method of more general application, such as that of Cain (ANALYST, 1911, 36, 475), must be used; but he considers that in routine work these more exact methods give no more useful information, whilst they consume more time.

G. C. J.

Rapid Method for Determination of Vanadium in Steels, Ores, etc., based on its Quantitative Inclusion by the Phosphomolybdate Precipitate. J. R. Cain and J. C. Hostetter. (*J. Ind. Eng. Chem.*, 1912, 4, 250.)—For steels containing vanadium, chromium, nickel, titanium, manganese, molybdenum, singly or in combination, an amount of drillings estimated to contain from 2 to 10 mgrms. of vanadium is dissolved in nitric acid (sp. gr. 1·135), of which about 20 to 25 c.c. is taken for each grm. of drillings. The solution is boiled until free from fumes, oxidised with permanganate solution ($1\frac{1}{2}$ per cent.), the manganese peroxide is dissolved by addition of sulphurous acid, and the solution again boiled until free from fumes. It is then nearly neutralised with ammonia (1 : 2), and a solution of sodium phosphate is added in such amount that there is at least ten times as much phosphorus as there is vanadium present. The solution is heated to boiling, and, after removal from the hot plate, ordinary molybdate reagent (0·05 to 0·06 grm. MoO_3 per c.c.) is added at the rate of at least 2 c.c. for each mgrm. of phosphorus present, and in no case less than 50 c.c. The mixture is agitated for a minute or so, the supernatant liquid decanted through an asbestos filter, and the precipitate washed three times by decantation with a hot solution of acid ammonium sulphate, made by adding 15 c.c. of ammonia (sp. gr. 0·9) and 25 c.c. of strong sulphuric acid to 1,000 c.c. of water. The last wash liquor is decanted off as completely as possible from the precipitate in the flask, and the filter is sucked dry. The filter is now connected to a small dry pump flask, and the small amount of precipitate on it is dissolved in hot concentrated sulphuric acid, which is then sucked through the filter and transferred to the flask containing the bulk of the precipitate, and the pump flask is rinsed out once with sulphuric acid. The total amount of acid used should be from 5 to 8 c.c. for every 10 mgrms. of phosphorus present. The contents of the flask are heated until solution takes place, a few drops of nitric acid (1 : 25) are added, and the solution is heated until it fumes strongly. It is then removed from the hot plate, and successive small additions of hydrogen peroxide solution are made, with vigorous shaking after each addition, until the solution assumes a deep brown colour, due to action on the molybdate. The flask is replaced on the hot plate and kept fuming for four or five minutes, when the brown colour gives place to a green or blue. The flask is covered and its contents cooled, and then poured into four times its volume of cold water and titrated at 70° to 80° C. against $\frac{N}{100}$ permanganate.

The function of the hydrogen peroxide is to reduce vanadium to the tetravalent state.

With steels containing tungsten, the only change necessary is to dissolve in *aqua regia*, dilute with hot water, filter off the tungstic acid, nearly neutralise with ammonia, and add 10 grms. of ammonium nitrate for every 100 c.c. of the final volume before precipitating as above described.

The behaviour towards solvents of a phosphomolybdate precipitate containing vanadium is very different from that of pure ammonium phosphomolybdate, and the choice of an acid solution of ammonium sulphate as wash liquor is based on experiments which are described in the paper, and which have some bearing on the estimation of phosphorus in steels containing vanadium.

The method can be extended to the estimation of as much as 30 mgrms. of vanadium, but the manipulation in such cases becomes inconvenient. From 2 to 10 mgrms. can be estimated with an error not exceeding 2 per cent., and the method permits of the concentration of the vanadium from 50 or 100 grms. of steel of low vanadium content, so that extremely small percentages can be determined with ease. A method is described whereby the vanadium may be separated as pentoxide in cases where there could be the slightest doubt of its being present at all. G. C. J.

New Method for Determination of Vanadium in Steel. D. J. Demorest. (*J. Ind. Eng. Chem.*, 1912, 4, 249-250.)—The method depends on the selective oxidation of ferrous sulphate in the presence of vanadyl sulphate by means of manganese dioxide. The vanadyl sulphate is then titrated by adding excess of permanganate, the excess permanganate being determined by means of sodium arsenite. The manganese dioxide should be sufficiently fine to pass through a 200-mesh sieve, and yet should settle in a beaker of water in thirty seconds (see next abstract). The borings (2 grms.) are dissolved in a mixture of 30 c.c. water and 12 c.c. sulphuric acid contained in a 500 c.c. flask. The iron is oxidised by addition of 1 c.c. of nitric acid, and the solution is boiled for a few minutes to remove nitrous fumes. It is then diluted with 30 c.c. of water, and excess of strong permanganate solution is added, and the solution boiled to completely oxidise all carbon. Ferrous sulphate is next added to reduce manganese dioxide, permanganate, chromate, and vanadate, and the solution again boiled to expel any possible nitrous fumes. The solution is diluted to about 250 c.c., and, after addition of $\frac{N}{10}$ permanganate until a pink colour persists, it is cooled. Ferrous sulphate is now added in amount at least sufficient to reduce all the chromic and vanadic acid, and any other reducible compound which may be present, but a large excess should be avoided. The solution is next shaken vigorously with about 1 grm. of manganese dioxide, and after two minutes a drop is tested with ferricyanide. The test is repeated at intervals of a minute, shaking all the while, until, usually in about five minutes, ferrous iron is found to be absent, when the shaking is continued for about another half minute, and the solution is then filtered through asbestos, using suction. The reduction of the iron is complete when the drop withdrawn does not instantly develop a blue colour when added to a drop of ferricyanide. A blue colour always develops in the course of a few seconds, due to the reduction of ferricyanide to ferrocyanide by the vanadyl sulphate. To the filtered solution standard permanganate is added from a burette until a pink tinge persists; 1 c.c. more is then added, and the excess of permanganate is titrated with sodium arsenite solution. The standard solutions employed correspond to 0·001 grm. vanadium per c.c. A blank determination must be run on a vanadium-free steel, the blank being generally about 0·7 mgrm. vanadium. Quantities of vanadium up to 7 mgrms. in 2 grms. of steel may be determined with an error not exceeding 0·1 mgrm., and the presence of ten times as much chromium is without influence on the results.

G. C. J.

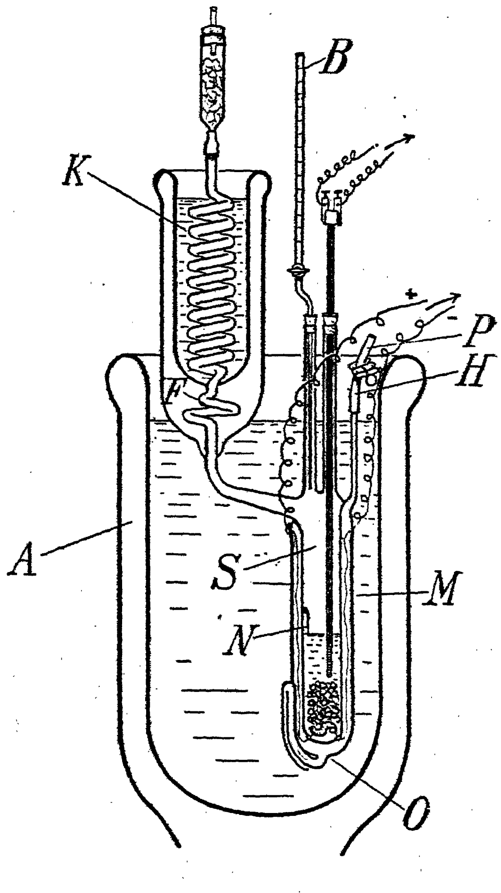
New Method for Determination of Vanadium. J. R. Cain and D. J. Demorest. (*J. Ind. Eng. Chem.*, 1912, 4, 256.)—J. R. Cain has found (*J. Ind. Eng. Chem.*, 1911, 3, 476) that manganese dioxide oxidises vanadyl compounds as well as ferrous compounds. This appears to conflict with the differential oxidation of iron by manganese dioxide on which Demorest's method of estimating vanadium (see preceding abstract) is based. Working together, the authors find that the difference in their results is solely due to the difference in grain of the dioxide each used. Very finely divided manganese dioxide oxidises vanadyl compounds completely, but when only of the fineness indicated in Demorest's paper, the velocity of this reaction is so small that its effect is inappreciable in the short time required for the quantitative oxidation of the iron. G. C. J.

APPARATUS, ETC.

Laboratory Centrifuge for Quantitative Separation and Estimation of Solids and Liquids. H. Zimmermann. (*Zeitsch. angew. Chem.* 1912, 25, 718-719.)—The centrifuge is arranged to carry four cylindrical copper beakers mounted on trunnions. The cylinders are of 100 to 150 c.c. capacity, and their height is about three times as great as their diameter. Their bottoms are cut away except for a narrow rim of metal which supports a removable perforated plate. In use a disc of felt is placed on the perforated plate, and a circular piece of wire gauze, mounted in a frame which exactly fits the cylinder, is placed on top of the felt. The apparatus has been specially constructed for determining the percentage of sugar crystals in raw sugars which contain syrup or molasses adhering to the crystals. Such raw sugar (50 grms.) is shaken with five times its weight of a saturated solution of cane-sugar in a pear-shaped bulb which is then connected by a rubber bung to one of the copper cylinders, and this to a filter pump flask. When the suction of the pump no longer extracts any liquid, the cylinder is capped with a rubber cap and whirled in the centrifuge for five minutes at 2,000 to 2,700 revolutions per minute. The contents of the cylinder are then pushed out, the perforated plate removed, and the sugar weighed with the felt and wire discs which have previously been tared. Usually a constant correction is made for syrup which persistently adheres to the crystals, but in any particular case the proper correction can be made by drying *in vacuo*, and assuming that for every part of moisture thus determined two parts of sugar were adhering to the crystals in the form of syrup. The whole centrifugal arrangement is surrounded by a case for safety and to avoid the projection of syrup, and when the apparatus is used for other purposes, where the liquor rather than the solid is the important constituent, the cylindrical beakers are provided with a collecting cup screwed on below the perforated false bottom. G. C. J.

Ebullioscopic Determinations at Low Temperatures (−36° to −83° C.).

E. Beckmann and W. Weber. (*Zeitsch. anorg. Chem.*, 1912, **74**, 297-309.)—The apparatus figured serves for ebullioscopic determinations with liquids such as hydrogen sulphide, chloride, bromide, and iodide as solvents. The greater part of the apparatus is seen immersed in a cooling liquid contained in a Weinhold-Dewar vessel of some 3 litres capacity. The central apparatus corresponds to the ordinary Beckmann apparatus, with arrangements for raising the temperature electrically and determining it by means of an electrical resistance thermometer. The boiling tube is surrounded by an air-jacket, which, however, is not sealed, but provided with an open syphon bend at the bottom, *O*, and a tube rising out of the bath, and adapted to be closed by a pinchcock, *P*, and rubber tube. When introducing the volatile solvent into the apparatus, suction is applied at *P*, so that the cooling liquid in the Dewar vessel may enter the air-bath and condense the solvent. When the level of the solvent reaches the glass point *N*, the liquid in the air-jacket is blown out by the use of a pressure ball. The bottom inch of the boiling tube is filled with garnets. *B* is a narrow burette for introduction of the second liquid—toluene in the authors' experiments. The temperature of the large cooling bath is maintained about 4° or 5° C. below the boiling-point of the solvent; that of the condenser, also contained in a Dewar vessel, about 10° to 15° C. below the boiling-point of the solvent. A lower temperature for the condenser is not permissible with hydrogen iodide, which solidifies some 16° below its boiling-point, and the boiling and freezing points of the other solvents also lie close together. In the case of hydrogen chloride, the cooling bath must consist of petroleum ether and liquid air, but for the other solvents a mixture of ether with carbon dioxide snow serves well, and with hydrogen sulphide a form of apparatus which dispenses with the 3-litre Dewar vessel may be



used. With the apparatus here figured, the authors have determined the molecular increase of the boiling-point of hydrogen sulphide as 6·3, hydrogen chloride 6·4, hydrogen bromide 15, and hydrogen iodide 26. These numbers are in good agreement with those deduced from the best attested values for the boiling-points and latent heats of vaporisation by the formula $K = \frac{0{\cdot}02\,T^2}{L}$. G. C. J.

Electrical Furnace for Studying the Course of Reactions under Great Pressure. F. Fischer and H. Ploetze. (*Zeitsch. anorg. Chem.*, 1912, 75, 1-9.)—A furnace is figured and described by means of which it is possible to heat substances to temperatures up to 900° C. under gas pressures as high as 100 atmospheres. The temperature at any particular point in the furnace can be determined within 20° C., but it is specially pointed out that other furnaces exist which give a larger area of uniform temperature. This furnace was specially designed to study the action on certain substances of oxygen at high temperatures and under high pressure. Essentially it consists of a porcelain tube, wound with platinum wire to conduct the heating current, and insulated by a considerable thickness of asbestos, etc., from an external casing constructed substantially of mild steel. The provision for the introduction of the current terminals, of the gas-leading tubes, etc., are necessarily somewhat elaborate, owing to the immense pressure, which tends to make all such connections leak. In use the whole apparatus is immersed in water, and one result of this is that there is a sharp temperature gradient between the middle and the ends of the tube. For certain purposes, however, this is an advantage, as it is possible to conduct simultaneously a number of experiments at different temperatures which may differ from one end to the other of a series by nearly 200° C. By means of this apparatus the authors have been able to prepare strontium peroxide, lead peroxide, and alkali peroxides by heating the corresponding oxides with oxygen under high pressures (*Zeitsch. anorg. Chem.*, 1912, 75, 10, 15, 30).
G. C. J.

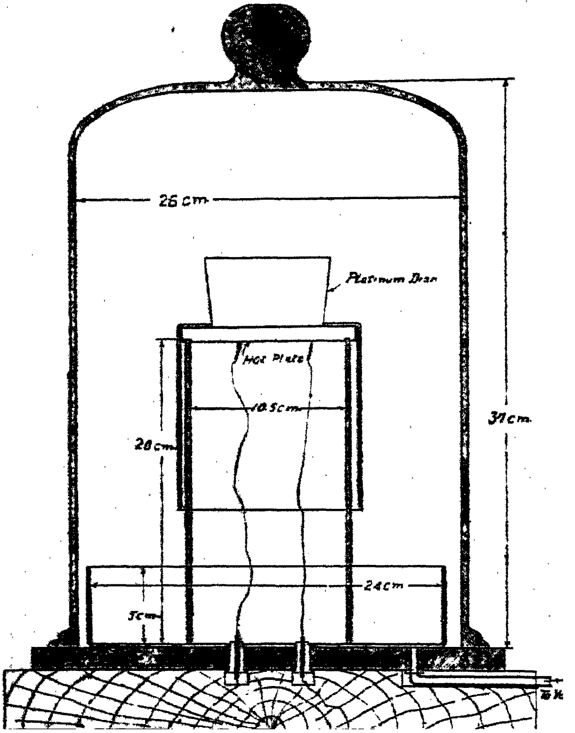
New Form of Extraction Thimble. P. A. Boeck. (*J. Ind. Eng. Chem.*, 1912, 4, 303-304.)—The thimbles are made of "alundum," the product of the fusion of bauxite in the electric furnace, in which process partial purification takes place, so that the resulting product is nearly pure fused alumina. In the manufacture of laboratory articles, the fused alumina is crushed, graded to uniform mesh, mixed with a suitable ceramic bonding material, moulded to the required shape, and fired in a porcelain kiln. The porosity of the bodies can be controlled by varying the size of the alundum grains or changing the kind or proportion of bonding material. Filtering thimbles and dishes of this material effect a great saving of time when substituted for paper thimbles, asbestos mats, and the like, in many laboratory operations. They are made by the Norton Company, Worcester, Mass., U.S.A. G. C. J.

New Forms of Gas Analysis Apparatus. G. A. Burrell. (*J. Ind. Eng. Chem.*, 1912, 4, 297-302.)—The author of this paper describes eleven pieces of apparatus in use by the U.S. Bureau of Mines. By means of one piece of apparatus it is possible to determine methane in mine air with an accuracy of 0·01 per cent. in ten minutes. After measuring, the gas is passed into a slow-combustion pipette containing an electrically heated platinum spiral and when combustion is complete—that is to say, after three or four minutes—the pipette is allowed to cool and the gas transferred back to the burette, and the contraction due to the burning of methane determined. The method, which is much quicker than Winkler's titration method, assumes the absence of other combustible gases in more than negligible traces, but this is often a safe assumption. A portable form of this apparatus is also figured, as well as one for the determination of methane and carbon dioxide. Another apparatus serves for the determination of small amounts of carbonic oxide by the iodic acid method. Considerable aid may be rendered in subduing mine fires by analysing samples of the atmosphere taken from behind stoppings, in order to determine whether the latter are sufficiently tight to exclude air, and a modified Orsat apparatus specially designed for this service is figured. Finally, there is a laboratory apparatus, and a portable modification of it, for the analysis of mixtures containing carbon dioxide, oxygen, ethylene, carbon monoxide, hydrogen, methane, and nitrogen. Special provision is made for the avoidance of the so-called capillary error which attaches to most ordinary forms of Orsat apparatus, and Babb's pipettes (*Eng. Soc. West. Pennsylvania*, 1904, **20**, 421) are used for cuprous chloride. G. C. J.

Marking of Porcelain Crucibles, etc. P. A. Yoder. (*Chem. Eng.*, 1912, **15**, 102-103.)—The author's platinum process for marking laboratory utensils of porcelain or silver-ware is as follows: The crucibles are cleaned by heating for half an hour with nitric acid and water (1 : 1). The parts of the apparatus to be marked are dipped into a hot gelatin size (5 per cent. solution of gelatin), and set aside to dry. When dried, the desired number is stamped on with rubber type with a solution of platinic chloride containing 12 to 15 per cent. of platinum. The pad may be made of six or eight folds of smooth linen or muslin a little larger than the type used, and is nearly saturated with a few drops of the platinum solution. After the numbers are dry, the crucibles are gently heated till reduction takes place, and the gelatin is burnt off, preferably in a muffle, and finally for one half-minute at the full temperature of a blast-lamp. The figures are made more prominent if burnished by means of a burnishing stone or of sea sand. The markings are resistant to single acids, but not to alkalis. Gold, and mixtures of gold and platinum, may be applied similarly. A. R. T.

New Silver Spiral for Use in Organic Elementary Analysis. A. H. Fiske. (*Ber.*, 1912, **45**, 870-871.)—In place of a spiral of silver foil or wire, the author recommends the use of a spiral of silver gauze, or better, because cheaper and equally efficient, a spiral of copper gauze plated with silver. After dipping in silver nitrate solution to give it a coating of silver, the gauze spiral is heated in a blowpipe-flame till the silver coating begins to show signs of melting. In certain cases it is convenient to plate the spiral only for half its length, thus obtaining a combined copper and silver spiral (*cf.* Zincke and Kegel, *Ber.*, 1890, **23**, 246). G. C. J.

New Apparatus for Vacuum Sublimation. G. W. Morey. (*J. Amer. Chem. Soc.*, 1912, **34**, 550-552.)—The apparatus for the sublimation of considerable quantities of organic compounds *in vacuo* consists of a bell-jar 26 cm. in diameter, accurately ground on a glass base, a large, shallow crystallising dish fitting inside the bell-jar and resting on the glass plate, and a glass cylinder, 10·5 cm. in diameter and 20 cm. high, cut from a piece of wide tubing, standing in the crystallising dish. This cylinder serves as a support for the electrical heating element of ordinary type, consisting of a heated iron plate; the top of the cylinder is loosely ground into the enamel on the bottom of the plate. The wires carrying the current enter through the bottom as shown, holes being bored in the crystallising dish, through which the rubber stoppers insulating the wires are inserted. It is best to have the wires in two parts, with connectors near the top to facilitate the removal of the heating element. The substance is contained in a flat-bottomed platinum dish or crystallising dish resting directly on the hot plate, and a deep crystallising dish with a hole cut out to receive the platinum dish is inverted over the hot plate to prevent the sublimed substance coming in contact with the iron. Most of the sublimed substance drops into the crystallising dish at the bottom, and the material comes in contact with nothing but glass and platinum. J. F. B.



REPORTS.

Investigations of Explosives used in Coal-Mines. C. Hall, W. O. Snelling, S. P. Howell, G. A. Burrell, C. E. Munroe. (*Bulletin* 15, *Bureau of Mines. Washington*, 1912.)—Numerous explosions of fire-damp in American coal-mines led to the foundation, in 1908, of the Pittsburgh testing station, where explosives could be examined, particularly for their behaviour in fiery mines. This volume contains: An introduction; a chapter on the nature of explosives; a simple treatment of thermochemistry, intended to enable manufacturers to control the explosion temperature—which is one of the factors of safety—of their products; remarks on the characteristics of the natural gas used in the testing gallery; and an account of the testing equipment of the station and modes of using it. This includes the ballistic pendulum, the gallery for gas, and coal-dust and gas; Mettegang recorder for determining rate of detonation; apparatus for determining photographically the flame length of explosives; apparatus for finding the length of air-gap over which explosion is communicated from one cartridge to another; impact machine, for sensitiveness; Bichel closed vessel, for explosion pressure and composition of explosive gases; calorimeter; and Trunzl lead block, for power. Results of tests on dynamites classed (by the Pittsburgh station) as "permissible" and black blasting powder are given; those on four samples of foreign "permitted" explosives show that the natural gas is more sensitive than the foreign pit gas used in test galleries, and would require dilution with air to give comparable results.

O. E. M.

Investigations of Fuse and Miners' Squibs. C. Hall and S. P. Howell. (*Technical Paper* 7, *Bureau of Mines. Washington,* 1912.)—Five classes of fuse are used in the United States, distinguished by their varying resistance to moisture. A standard rate of burning for all fuse should be established. Six samples were examined for nature of covering, quantity of powder per foot, size of grain and composition of powder, and two for gaseous products of combustion. An improved sample with a wrapper treated with silicate of soda, and containing 0·63 grm. of powder per foot, gave only one-third as much gas as an untreated fuse with 1·08 grms. per foot; it gave much less sulphuretted hydrogen and carbon monoxide—an important feature in confined spaces. A fuse containing 0·63 grm. per foot ignited black blasting powder with certainty, and was safe in gas mixtures. The safety in gas mixtures increased with diminishing size of powder core. A fuse suitable for use in fiery mines should have a uniform core, an envelope strong enough to prevent bursting, pliable, and waterproof, a safe and certain means of ignition, a certain action, and a uniform rate of burning. Miners' squibs are excluded from use in fiery mines on account of their spit. O. E. M.

REVIEWS.

CHURCH'S LABORATORY GUIDE. Revised by EDWARD KINCH. 9th Edition. Gurney and Jackson. London. Price 6s. 6d. net.

The demand for a new edition of this well-known work, five years after publication of the eighth, bears testimony to its popularity among those for whom it is written. The division into three parts, dealing with Chemical Manipulation, Qualitative Analysis, and Quantitative Analysis, respectively, is maintained, and is to be highly commended, the exercises comprised in the first part being so selected as to encourage the student, early in his course, to realise that the pursuit of chemistry will assist him to a better understanding of agricultural products and processes. The section which gives individuality to the book and which calls for most praise is that devoted to Quantitative Analysis, as it contains much information to which an elementary student has not ready access, and deals in a comprehensive manner with manures, soils, waters, and foods.

It is to be regretted, however, that an otherwise useful manual should be marred by some obvious blemishes of style and treatment. The violent fluctuations in mood and tense throughout the opening pages of Part I. set a poor example for the student to follow in his own notes. The expression to "syringe out" a solid from one vessel to another is so unfamiliar that one automatically searches for a syringe in the list of apparatus required for the experiment. Such barbarities as "arseniosum," "arsenicum," "antimoniosum" and "stannicum," jostling copper without a word of explanation, can only mystify a beginner, whilst the unvarnished empiricism of the "sugar test" is deplorable. Although the "sugar test" is used on pp. 53, 54, 55, 59, 292, 295, and 301, instructions for its preparation being given on p. 86, the name Fehling is first encountered in small print on p. 303, and the only index reference to Fehling's solution is p. 86, where the name does not occur, and its connection with the "sugar test" is consequently lost. M. O. FORSTER.

HUILES ET GRAISSES VÉGÉTALES COMESTIBLES. Par G. HALPHEN. Librairie Polytechnique Ch. Béranger. Paris et Liège. 1912. Price 8s. net.

Concerning this book (which forms one of a series of practical manuals of chemical analysis published under the direction of MM. Bordas and Roux) one can have nothing to add but praise, and, in fact, the expectations raised by the name of such a well-known investigator as Halphen are fully realised.

Ostensibly the book deals with seven vegetable oils which are most commonly met with—namely, olive, arachis, cotton, poppy-seed, walnut, sesame, and cocoanut oils—but, on account of the minute details of the various possible adulterants of these oils, a very wide field dealing with the characteristics and properties of many other oils is covered. The method of treatment of the subject, and particularly the details of official methods, are different in many ways to those with which the English analyst is likely to be familiar; but this is only to be expected in a book written particularly for those engaged in the subject as it presents itself in the country of publication. In spite of this, the work is full of suggestions to the analyst, and there are a number of methods detailed and discussed to which many will be quite strangers.

One of the leading features of the book is the stress laid by the author on the use of colour tests in the detection of adulteration, and he defends himself both ably and convincingly against the attacks which have often been made upon his position.

The only possible exception that can be taken to the manner of treatment is, after all, an exception which really redounds to the credit and knowledge of the author, and that is to the somewhat full treatment of the various subjects which might possibly leave the casual investigator in a rather undecided state of mind, but which to the expert will be a source of advantage and assistance. This is exemplified by the fact that, out of about 450 pages, nearly 230 are taken up with the discussion and description of the method of determination of the various constants, etc. The book requires close reading, as in quite an incidental manner most useful hints with regard to technical manipulation, such as, in particular, the method given for incinerating fats, on p. 38, and for the estimation of total foreign matter, on p. 34, and many others are inserted, and the author has again brought to the front and supported with all the weight of his authority several useful tests which have, on account of adverse criticism, been allowed to lapse into an obscurity which was certainly not deserved.

Though the work abounds in tables of figures, it is extremely free from manifest errors, and, in fact, the only one which has been noticed of sufficient seriousness to lead the analyst astray is the transposition of the words "cholestèrine" and "phytostèrine" throughout an entire section on p. 221.

The author has also included a useful and sensible section on the production of rancidity.

The last section of the book contains an appendix dealing with the regulations in force in the different countries, together with copies of the various circulars issued to the official chemists by the French Minister of Agriculture, as well as some definitions of oils as set forth in the findings of the First International Congress for the Suppression of Adulteration, Geneva, 1908.

In conclusion, it may be said that this book is well worthy of the attention of those engaged in the examination of edible oils and fats.

E. Richards Bolton.

NEW BOOKS.

CHEMICAL WORKS: THEIR DESIGN AND EQUIPMENT. By S. S. DYSON and S. S. CLARKSON. London: Scott, Greenwood and Son. 1912. Price 21s. net.

Octavo volume, containing 204 pages of subject matter, with 80 illustrations and 9 plates, and an alphabetical index. The text is classified under the following heads: I. Choice of Site. II. Materials used in Construction. III. First Principles in Laying Out of Works. IV. The Power-House. V. Sulphuric Acid Plant. VI. Hydrochloric Acid Plant. VII. Nitric Acid Plant. VIII. Notes on High Explosives Plant. IX. Sulphate of Ammonia Plant. X. Artificial Manure Plant. XI. General Plant. Appendixes: I. The Chemical Engineer. II. The Saw-Mill and Box-making Department. III. The Alkali, etc., Works Regulation Act. IV. "Welfare Work" or "Prosperity Sharing."

QUALITATIVE ORGANIC ANALYSIS. By F. B. THOLE. London: Methuen and Co. 1912. Price 1s. 6d. net.

The contents comprise—I. Common Operations in Practical Organic Chemistry. II. Determination of Melting and Boiling Points. III. Preliminary Tests. IV. Identification of Aliphatic Compounds. V. Identification of Carbocyclic and Heterocyclic Compounds. VI. Separation and Identification of Mixtures. Appendixes. Index.

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INSTITUTE OF CHEMISTRY.

PASS LIST: APRIL EXAMINATIONS, 1912.

OF fifteen candidates who presented themselves for the Intermediate Examination, six passed: L. M. Clark, A. L. Davidson, F. C. Guthrie, B.A. (Cantab.), J. T. Janson, B.Sc. (Lond.), T. S. Jones, B.Sc. (Lond.), and D. A. Legg. Of twenty-five candidates who presented themselves for the Final Examination, fifteen passed. In the branch of Mineral Chemistry: A. M. Bailey, H. W. Gill, B.Sc. (Lond.), D. McDonald, B.Sc. (Lond.), A. H. Maude, and C. R. Robson; in the branch of Physical Chemistry: F. W. Atack, B.Sc. Tech. (Manc.); in the branch of Organic Chemistry: G. McL. Carruthers, R. B. Croad, J. R. Douglas, A.R.C.Sc.I., A. J. Hale, B.Sc. (Lond.), D. Hamilton, J. Porter, and A. Rayner, B.Sc. (Lond.); in the branch of the Chemistry of Food and Drugs, and of Water: L. E. Campbell, B.Sc. (Lond.), and E. Hill, A.R.C.Sc. (Lond.).

JULY, 1912. Vol. XXXVII., No. 436.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The monthly meeting of the Society was held on Wednesday evening, June 5, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. S. W. Collins, B.Sc., F.I.C., and C. B. Lessner, were read for the second time.

Messrs. H. T. Cranfield, E. G. Jones, M.Sc., F.I.C., and W. H. Roberts, M.Sc., F.I.C., were elected members of the Society.

The following papers were read: "The Composition of Milk," by H. Droop Richmond, F.I.C.; "The Estimation of Dirt in Milk," by W. Foulkes Lowe, A.R.S.M., F.I.C.; "On the Application of Adsorption to the Detection and Separation of Certain Dyes," by A. Chaston Chapman, F.I.C., and Alfred Siebold, F.I.C.; "A New Method for the Detection and Estimation of Small Quantities of Nitrous Acid," by E. Holl Miller; "A Further Note on the Use of Methylene Blue as an Indicator in Iodimetric Titrations," by Frank Sturdy Sinnatt; and "The Estimation of Nitric and Nitrous Acids in Acetic Acid Solution—The Stability of Nitric Acid in Acetic Acid Solution," by Kennedy J. P. Orton, M.A., Ph.D., F.I.C.

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THE CONSTITUENTS OF OIL OF SAVIN.

By J. WATSON AGNEW F.I.C., and ROBIN B. CROAD.

(*Read at the Meeting, May* 1, 1912.)

Several samples of oil of savin have been analysed with the object of obtaining pure sabinene, and the following paper is an account of the method used to obtain the terpene, with a description of the various fractions obtained from a pure specimen of oil of savin.*

The oil was first of all hydrolysed by boiling under a reflux condenser with alcoholic potash for a week, the amount of caustic potash used being the theoretical quantity required to hydrolyse sabinol acetate (on the assumption that this ester were the sole constituent of the oil), since smaller quantities of potash always leave a large quantity of ester unhydrolysed. When the hydrolysis was finished, carbon dioxide was passed through the liquid until saturated. The solution was then filtered, and the filtrate distilled in a current of steam, the oily distillate separated and dried over anhydrous sodium sulphate. The dry oil was then fractionally distilled, using a six-bulb fractionating column. The flasks for each fraction should be closed, since the oil rapidly absorbs moisture from the air, and this interferes with the subsequent refractionation.

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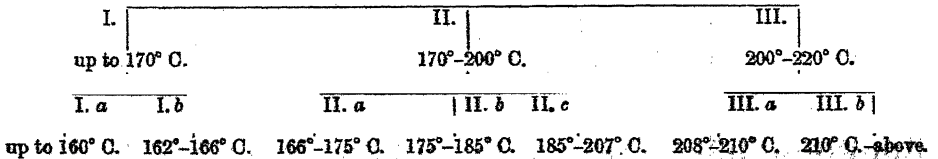
* This oil was obtained from Schimmel, who guaranteed it to be a genuine sample of oil of savin. The sample contained very little pinene, but other samples which we examined from other sources had evidently been mixed with oil of turpentine, as the amount of pinene was very high, while the sabinene was correspondingly low.

The oil was fractionated according to the following scheme:

Products of hydrolysis of savin oil.



Each of these fractions was then separately redistilled at a slow rate.

DESCRIPTION OF FRACTIONS OBTAINED.

I. *a*. In most of the samples examined this fraction was large in comparison to I. *b*, though in one case the amount of I. *b* was very large, whilst I. *a* was an exceedingly small fraction. This fraction was undoubtedly pinene, since on oxidation with mercuric acetate solution, a mixture of sobrerol and 8-hydroxycarvotanacetone was obtained. The presence of these two oxidation products of pinene was confirmed by the formation from the original sobrerol (m.-pt. 131° C.), of sobrerol dibromide (m.-pt. 131°–132° C.), and pinol dibromide (m.-pt. 94° C.). The sobrerol dibromide was analysed with the following result:

Br calculated for sobrerol dibromide = 48·48 per cent.
Br found ... ... = 48·27 „

The semicarbazone of the hydroxyketone was also prepared, and was found to melt at 175° C. This agrees with what already has been published (Henderson and Agnew, *J. Chem. Soc.*, 1908, **95**, 289), and the yields from pure pinene and from this fraction are comparable.

I. *b*. As already stated, this fraction was only obtained in large quantity in the case of one sample, and consisted of the terpene sabinene. That this fraction is sabinene was shown by shaking a small quantity with dilute sulphuric acid for about two weeks, when a terpin (m.-pt. 137° C.) and a terpineol (b.-pt. 209–212° C.) were isolated from the solution (Wallach, *Ber.*, 1907, **40**, 585). As these were obtained pure it is practically certain that no pinene was present, or crystals of a terpin hydrate (m.-pt. 120° C.) would have separated out. This was further confirmed by preparing the nitrosochloride according to the method used for pinene nitrosochloride (Wallach, *Annalen*, 1889, **253**, 250), the only resulting product being a sticky, green oil, with no trace of the solid pinene nitrosochloride. The formation of this green oil is in accordance with the observations of some other workers. The constants of the pure sabinene were as follows: b.-pt. 162° to 166° (chiefly 164° C.); sp. gr. 20° C. = 0·8468; $[\alpha]_{D\ 15^\circ\ C.} = -42{\cdot}5$.

It will be noticed that the oil was lævo-rotatory, which is contrary to the published results of other workers, who have only noted the dextro form. Semmler (*Ber.*, 1900, **33**, 1464) records a rotation +63°, and Wallach (*Ber.*, 1907, **40**, 587) a rotation of +67·5° $[\alpha]_D = 80{\cdot}17$. The sp. gr. is also somewhat higher than the figures usually given (Wallach = ·8420, and Semmler = ·840).

These two fractions, I. *a* and I. *b*, can be tested by adding 2 to 3 c.c. to a saturated solution of mercuric acetate in a Nessler tube. In the case of sabinene, oxidation commences at once, and there is a large precipitate of mercurous acetate at the end of fifteen minutes. In the case of pinene there is no oxidation until at least one hour has elapsed.

II. *a*. On careful refractionation this portion splits up into two fractions with the same boiling-points and properties as I. *b* and II. *b*.

II. *b*. This fraction, according to Fromm (*Ber.*, 1898, **31**, 2025), consists of terpinene, and this was confirmed by the ready formation of the nitrosite (m.-pt. 155° to 157° C.) in good quantity (Wallach, *Annalen*, 1887, **239**, 36).

II. *c*. This fraction was redistilled, and the lower and higher fractions were added to II. *b* and III. *a* respectively.

III. *a*. This fraction consisted of the alcohol sabinol, the constants of which were found to be as follows: b.-pt. 208° to 209° C.; sp. gr. 15° C. 0·9391; $[\alpha]_{D\ 15^\circ\ C.} = +17{\cdot}04$. The rotation of this sabinol is much higher than any we have observed in published results.

III. *b*. The residue from this fractionation consists chiefly of unhydrolysed esters.

The solution left in the flask after the original steam distillation contained the potassium salts of the acids, originally present in the esters. This solution was extracted with ether to remove resin and then evaporated down to dryness. The purified potassium salts thus obtained were acidified with dilute sulphuric acid and extracted with ether. The residue, after evaporating off the ether, was found to be a mixture of formic and acetic acids, with a small quantity of another acid, which melted at 85° C. This acid is saturated and is colourless when first isolated, but turns brown in the air. The presence of formic and acetic acids in oil of savin has been already noted, and the quantity of this third acid was too small to permit of our fully identifying it. The following is a table showing the percentage yields:

1. Resin from steam distillation	31·0
2. First runnings 150° to 160° C.	1·7
3. Sabinene 162° to 166° C.	16·0
4. Terpinene fraction 175° to 185° C.	5·3
5. Sabinol 208° to 209° C.	17·0
6. Residue 210° C. upwards	16·0
7. Acids—calculated from amount of sabinol ...	7·0

The percentage of resin is high owing to the difficulty of entirely freeing from water. According to Semmler (*Ber.*, 1900, **33**, 1463), a yield of 30 per cent. of sabinene was obtained by fractionating the crude oil. Altogether we have examined about 15 lbs. of oil of savin in different samples, and the above results are those of the best sample, which gives 16 per cent. In some cases the amount of sabinene was exceedingly low, and the amount of pinene correspondingly high.

In conclusion we desire to express our thanks to Professor G. G. Henderson for the interest he has shown in this work and for many valuable suggestions.

Department of Chemistry,
Royal Technical College, Glasgow.

Discussion.

Mr. H. E. Burgess said that he had examined a large number of samples of savin oil, and had never found any fraction that was not dextro-rotatory. It would seem that the sample worked upon by the authors must have been either inactive or lævo-rotatory, and therefore, as far as his experience went, abnormal.

Mr. Raymond Ross suggested that the authors might with advantage add to their communication the refractive indices of the various fractions. For the determination of the optical activity a considerable quantity of material was required, and the refractive index—which, as well as the boiling-point, could be determined with a few drops—would be more useful in actual practice, where the quantity of material available was often very small.

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THE COMPOSITION OF MILK.

By H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, June* 5, 1912.)

The present communication deals with the results obtained during 1911 in the Aylesbury Dairy Company's Laboratory.

In Table I. the mean composition of 19,997 samples of milk as received from the farms is given. This series is selected, as it contains the largest number of samples, and is therefore the most nearly representative; the morning and evening milks are averaged separately. So far as the mean composition is concerned, it is confirmed by four other series, consisting of 11,269, 1,472, 2,908, and 1,836 samples respectively.

The average percentage of fat (3·71) is slightly below that found last year, and this was principally due to a falling off in the last months of the year. As is almost universally found, the lowest percentages of fat occurred in May and June and the highest in October and November. The evening milk is practically always richer than the morning milk, but the average difference found (0·30 per cent.) is slightly less than usual, and the slightly smaller amount of fat found in 1911 is due to that in the evening milk being less.

TABLE I.

Average Composition of Milk during 1911.

Month.	Morning Milk.				Evening Milk.				Mean.			
	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.
		%	%	%		%	%	%		%	%	%
January ...	1·0323	12·64	3·68	8·96	1·0321	12·86	3·91	8·95	1·0322	12·75	3·80	8·95
February ..	1·0325	12·55	3·57	8·98	1·0322	12·79	3·83	8·96	1·0324	12·67	3·70	8·97
March ...	1·0324	12·41	3·49	8·92	1·0321	12·66	3·76	8·90	1·0322	12·53	3·62	8·91
April	1·0321	12·31	3·48	8·83	1·0318	12·59	3·77	8·82	1·0319	12·45	3·62	8·83
May	1·0323	12·12	3·26	8·86	1·0319	12·51	3·68	8·83	1·0321	12·32	3·47	8·85
June	1·0323	12·09	3·24	8·85	1·0316	12·42	3·64	8·78	1·0320	12·26	3·44	8·82
July	1·0316	12·17	3·44	8·73	1·0309	12·35	3·74	8·61	1·0313	12·26	3·59	8·67
August ...	1·0309	12·13	3·54	8·59	1·0304	12·42	3·90	8·52	1·0306	12·27	3·72	8·55
September ...	1·0311	12·35	3·69	8·66	1·0306	12·68	4·07	8·61	1·0309	12·51	3·88	8·63
October ...	1·0315	12·52	3·76	8·76	1·0312	12·82	4·07	8·75	1·0313	12·67	3·91	8·76
November ...	1·0316	12·65	3·83	8·82	1·0314	12·85	4·05	8·80	1·0315	12·75	3·94	8·81
December .	1·0318	12·54	3·72	8·82	1·0315	12·69	3·88	8·81	1·0317	12·61	3·80	8·81
Average ...	1·0319	12·37	3·56	8·81	1·0315	12·64	3·86	8·78	1·0317	12·51	3·71	8·80

In continuation of the tables given in former years, I give the percentages of the morning milks falling below 3 per cent. in April, May, and June.

TABLE II.

Month.	2·9 to 3·0.	2·8 to 2·9.	2·7 to 2·8.	Below 2·7.
April	0·8	0·2	0·2	—
May	4·0	2·1	0·5	0·2
June	5·5	0·5	0·5	0·2

In the evening and morning milks during the other months of the year samples containing less than 3 per cent. of fat were practically absent.

The year 1911 was remarkable for the prolonged drought during the summer months, and not only was the quantity of milk influenced by the deficient herbage, but also the quality. It is usual to find that the solids-not-fat are lower in July, August, and September than during the other months, but during 1911 a greater deficiency than usual was observed.

The table below shows the percentages of the milks falling between the percentages of solids-not-fat given for these three months:

TABLE III.

Month.	8·4 to 8·5.	8·3 to 8·4.	8·2 to 8·3.	Below 8·2.	Total.
July	6·5	1·1	0·2	0·1	7·9
August ...	19·9	6·5	0·4	0·2	27·0
September ...	10·4	2·6	0·1	0·1	13·2

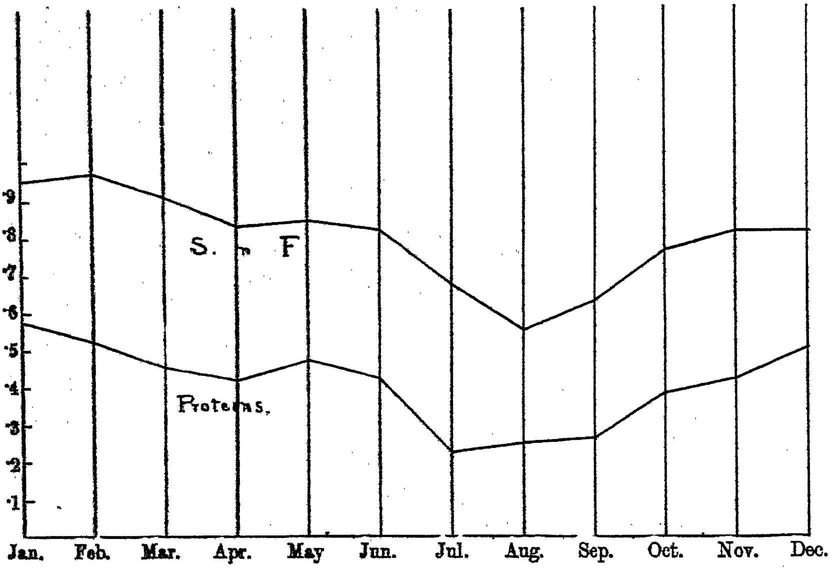
During the other months of the year very few samples fell below 8·5 per cent. of solids-not-fat.

In order to see to what constituent of the solids-not-fat the deficiency was due, the following table has been prepared. This gives the mean percentages of solids-not-fat, the mean percentage of proteins determined from the aldehyde figure, the percentage of ash determined directly, and the percentage of milk-sugar by difference. Although the number of protein and ash determinations is very much smaller than that of the solids-not-fat, the average figure is probably sufficiently close to the true value to render a comparison possible.

TABLE IV.

Month.	Solids-not-Fat.	Milk-Sugar.	Proteins.	Ash.
January	8·95	4·62	3·57	0·76
February	8·97	4·70	3·52	0·75
March	8·91	4·72	3·45	0·74
April	8·83	4·66	3·42	0·75
May	8·85	4·64	3·47	0·74
June	8·82	4·68	3·42	0·74
July	8·67	4·69	3·23	0·75
August	8·55	4·59	3·25	0·71
September ...	8·63	4·63	3·26	0·74
October	8·76	4·63	3·38	0·75
November ...	8·81	4·63	3·42	0·76
December ...	8·81	4·56	3·50	0·75

It is seen from the above figures that the deficiency in solids-not-fat during July, August, and September is due principally to a deficiency in proteins, and this is shown in the accompanying curve.



In conclusion, I wish to express my indebtedness to Mr. H. C. Huish, to whom the bulk of the above figures are due.

DISCUSSION.

Mr. R. Ross asked whether Mr. Richmond found the proportions of 13 : 9 : 2 for milk sugar, proteins, and ash respectively, still to hold good for normal milk. In the case of several recent samples taken under the Sale of Food and Drugs Act, he (Mr. Ross) had found deficiency in non-fatty solids to be due to deficiency in milk sugar, and he had accordingly reported the samples to be not adulterated, but abnormal. He should like to know what were the intervals between the times of milking.

Dr. VOELCKER expressed some surprise that, in an exceptionally dry year like 1911, the quality of the milk was lower, and not higher, than in the previous year. He referred incidentally to the unreliability of the results shown by cows when milked in the showyard for purposes of competition, instancing a recent case, which had been commented upon in the Press, in which a Jersey cow, under such conditions, had yielded milk containing only 2·75 per cent. of fat. From subsequent inquiries it appeared that the cow in question was of an unusually nervous temperament, and that the poorness of her milk on that particular occasion was due entirely to nervous disturbance caused by the strange conditions. He instanced this as showing how much better it was to rely upon such statistics as Mr. Richmond had put forward, than on "showyard" results.

Mr. C. REVIS said that the results of his own analyses during 1911 were similar to those obtained by Mr. Richmond, but he was not quite satisfied that an average of the results of such analyses exactly represented the state of the case. Some three or four years ago he had abandoned the calculation of such averages, and, instead, had adopted the plan of analysing samples drawn at intervals as the milk was being measured out from the mixing vessel, through which would pass some 4,000 gallons twice a day. In this way one eliminated such errors as would be incidental to calculations based on separate samples from about 250 different farmers which this represented. During the two months in question, the non-fatty solids in this quantity of milk were below 8·45 in eleven cases, being in one case as low as 8·34. He had never before met with such low figures in mixed milk, and he thought that Mr. Richmond had not laid sufficient stress on the profound effect of last year's drought. The effect of the drought was felt for some months, and the slightly lower average percentage of fat found in 1911 was no doubt attributable to the almost entire failure of proper feeding-stuffs towards the end of the year.

Mr. A. E. PARKES asked whether Mr. Richmond considered it to be possible, by determining the percentages of milk sugar and proteins, to ascertain whether a sample had been artificially prepared with milk powder, or was a mixture of fresh milk and this product. During last year's drought a considerable quantity of milk had been made up in this way, and he believed that in some districts the practice was still going on.

Mr. L. M. NASH said that, in a large butter factory, it was found that the quantity of milk required for the making of a given quantity of butter was almost exactly the same in 1911 as in 1910, while in a casein factory it was found that the percentage of casein obtained from the milk during 1911 was lower than usual, Mr. J. Evans asked whether any of Mr. Richmond's samples came from the North of England.

Mr. J. Connah said that he had been under the impression that the contracts under which farmers supplied the large dairy companies included a stipulation that the milk should not be below a certain minimum standard of composition, any milk below that standard being rejected. He should like to hear whether Mr. Richmond's averages included such rejected samples or not.

Mr. Richmond, in reply, said that the proportions of 13 : 9 : 2 for milk sugar, proteins, and ash (which were originally laid down by Dr. Vieth) still held good. This was, indeed, according to his experience, the closest approximation to the average that could be obtained in round figures, and he was still strongly of opinion that any wide departure from the proportions of 13 : 9 : 2 indicated that the milk was abnormal. As to the times of milking, these varied considerably in different districts, and were, practically speaking, determined by the train service. On going into this question some little time ago, he had found that on the average there was an interval of 13·2 hours from the evening milking to the morning milking, and of 10·8 hours from the morning milking to the evening milking. He did not quite understand why Dr. Voelcker expected the quality of the milk to be higher in a dry season, unless it was because of the smaller yield. When the quantity was checked by such a cause as a fall in temperature, the resulting decrease in the total metabolic change would not be accompanied by a corresponding decrease in the fat metabolism, and consequently the proportion of fat in the milk would be greater; but when the decrease in quantity was due to a decrease in the total amount of actual nourishment, one would expect a falling off in quality as well. He entirely agreed with Dr. Voelcker that results obtained at shows could not be regarded as representing the normal composition of milk. It was a very good plan to take average samples from large quantities of milk. He had followed it himself, and his experience had been similar to that of Mr. Revis, though the quantities sampled were not so large as in Mr. Revis's case. The question raised by Mr. Parkes was a very difficult one, more especially when the sample consisted only partially of milk made up with milk powder. At present he did not see how it was to be solved. The most northerly district represented among the samples was Cheshire. With regard to Mr. Connah's remarks, the practice was to name in the contract a certain standard of composition, to which the farmer was expected to conform as nearly as possible. As, however, the farmer had no real control over the percentages of fat and of non-fatty solids yielded by the cows, milk falling below the contract standard would not necessarily be rejected, unless the deficiency was very serious, and even then the milk that was rejected would be used for making cream, and the samples would be included in the average unless there was evidence of adulteration. As far as the non-fatty solids were concerned, very low percentages would of course be rejected, but not percentages between, say, 8·0 and 8·5, unless further examination indicated the presence of added water. Low percentages of fat would not lead to rejection unless there was some evidence—such, for example, as the arrival of the churn unsealed, with a deficiency in the quantity of milk—to show that cream had been removed.

THE DETERMINATION OF NITRIC AND NITROUS ACIDS IN ACETIC ACID SOLUTION. THE STABILITY OF NITRIC ACID IN ACETIC ACID SOLUTION.

By KENNEDY J. P. ORTON, M.A., Ph.D., F.I.C., and WILLIAM HERBERT GRAY.

Although numerous papers on the determination of nitric and nitrous acids and their salts have been published, the determination of these acids in acetic acid solution has not been investigated. The inquiry arose from the need of knowing, for the purpose of experiments on the nitration of aromatic compounds, whether nitric acid was reduced by acetic acid. For these experiments very dilute solutions—not greater than 1 per cent.—of nitric acid were required. This fact was the objection to the use of the Kjeldahl-Gunning-Jodlbauer method, which would be applicable to more concentrated solutions. For example, it was used with success by Pictet and Khotinsky (*Ber.*, 1910, **40**, 1163) in the analysis of acetyl nitrate.

The method here described is based on the fact that all the nitric acid in acetic acid solution is retained as potassium nitrate if the solution is evaporated to dryness on the water-bath with a slight excess of potassium carbonate; any nitrous acid is destroyed under this treatment. The potassium nitrate, which is mixed with some acetate, is determined by the Lunge process. The nitrous acid can be best determined by Raschig's permanganate method (*Ber.*, 1905, **35**, 3911). Nitric acid is remarkably stable in acetic acid solution, and can be kept of constant concentration for many weeks. If nitrous acid (oxides of nitrogen) is present, the major part is gradually oxidised to nitric acid, the solution becoming nearly colourless. Solutions in 50 per cent. acetic acid showed similar behaviour. Reduction of the nitric acid does not occur even after prolonged exposure to diffused light, but any colour due to oxides of nitrogen originally present gradually fades, the concentration of the nitric acid somewhat increasing. This remarkable behaviour will be further investigated.

Determination of Nitrous Acid in Acetic Acid Solution.

All the well-known methods of analysis were tested. The problem resolved itself into ascertaining which of these could be used in a medium containing about 10 per cent. acetic acid, as that was practically the maximum dilution attainable in any but the permanganate method on diluting the original glacial acetic acid medium. Further, the method should be accurate in the presence of the relatively very large excess of nitric acid.

(*a*) The tintometric method with *m*-phenylenediamine failed completely. Compared with aqueous solutions of nitrite, solutions in dilute acetic acid (5 to 40 per cent.) showed paler tints. The discrepancy is progressively greater as the proportion of acetic acid is increased. It should be noted that the mere addition of acetic acid after diazotisation does not in itself affect the tint.

(*b*) The Dunstan-Dymond method. In the presence of small quantities of acetic acid (5 to 10 per cent.) this method gives high results. Moreover, in the very dilute solutions which we required, the manipulative difficulties are excessive.

(*c*) The method devised by Maderna and Coffetti (*Gazetta*, 1907, **37**, 595), which is based on the reduction in a Lunge nitrometer of nitrous acid to nitric oxide by potassium ferrocyanide and citric or acetic acid—an environment in which nitric acid is stable—was found to give excellent results in the analysis of fuming nitric acid, which was neutralised before introduction into the nitrometer, with slight excess of sodium hydroxide. The solubility of the nitric oxide in the more concentrated acetic acids, which were unavoidable in this method, rendered it inaccurate.

(*d*) In Raschig's method we found a perfectly satisfactory means of estimating minute quantities of nitrous acid in acetic acid solution. The following procedure was finally adopted :

A given volume of the acetic acid solution, 10 to 30 c.c., was added drop by drop, over a period of four minutes, to a mixture of about 20 c.c. of $\frac{N}{10}$ permanganate, 5·5 c.c. of 10 per cent. sulphuric acid and 320 c.c. of water. The permanganate should be about double that required for the oxidation of the nitrous acid; the sulphuric acid is nearly the minimum quantity required for this dilution; the nitrous acid, when added to the mixture, should not be below a dilution of 1 in 20,000. The mixture is allowed to stand for fifteen minutes, and then excess (about 8 to 9 c.c. of a 2·5 per cent. solution) of potassium iodide introduced, and the iodine titrated with $\frac{N}{10}$ of thiosulphate. The time of addition of the acetic acid solution could be considerably shortened without materially affecting the accuracy, but the fifteen minutes' standing is a minimum. The following numbers illustrate the results of determinations of nitrous acid in fuming nitric acids :

Percentage of Acetic Acid in the Permanganate Mixture.	C.c. of $\frac{N}{10}$ $KMnO_4$ used in oxidising Nitrous Acid.	Grms. of HNO_2 in 100 c.c. of fuming Nitric Acid.
5	8·38	1·97
5	8·48	1·99
10	8·44	1·98
10	8·50	1·99

THE DETERMINATION OF NITRIC ACID IN ACETIC ACID SOLUTION.

The procedure we adopted is as follows : To an aliquot part of the acetic acid solution, which is placed in a glass dish, covered by a clock-glass, a slight excess (5 to 25 per cent.) of $\frac{N}{1}$ potassium carbonate solution is cautiously added. The liquid is then evaporated on the water-bath until no odour of acetic acid remains, the clock-glass being left on the dish. The slightly moist solid is then transferred by a spatula to the cup of a Lunge nitrometer. The spatula is then washed with 0·3 c.c. of water into the cup, and the liquid drawn into the tube. Five c.c. of pure concentrated sulphuric acid (96·6 per cent.) are poured into the glass dish, when the all but negligible residue is dissolved, and then introduced into the nitrometer. It was found by trial that 4·5 c.c. of the 5 c.c. of the sulphuric acid used reached the nitrometer tube. This proportion of water and acid gave a concentration of 93·2 per cent. in the nitrometer. In shaking, etc., we followed the directions which Marqueyrol and Florentin ANALYST, 1911, 36, 243) recommend in their recent critical study of the Lunge method. It was found that, after two of the shakings described by these authors, occupying one minute each, the maximum volume of gas was obtained. In calculating the results, the nitric oxide absorbed by the sulphuric acid, as given by Lunge, was allowed for, namely, 0·15 c.c. for 5 c.c. of sulphuric acid.

This method was tested by blank experiments (*a*) with potassium nitrate, (*b*) with mixtures of potassium nitrate and nitrite.

(*a*) On evaporating pure potassium nitrate with acetic acid on the water-bath, no nitric acid was lost, since no acetic acid was recognisable in the residue by any of the usual qualitative tests. The following numbers illustrate the results:

C.c. of Acetic Acid.	KNO_3 used.	C.c. of NO.	KNO_3 found.	Percentage Error.
5	0·1188	25·81	0·1180	0·7
5	0·1192	25·94	0·1181	0·95
5	0·1185	25·84	0·1175	0·80

(*b*) Mixtures of potassium nitrate and nitrite were evaporated with glacial acetic acid. The residue gave no trace of the reactions of nitrous acid. Quantitative determinations of the nitrate showed that no nitrite had been oxidised to nitrate.

C.c. of Acetic Acid.	Potassium Nitrite as a Percentage of Nitrate.	KNO_3 used.	KNO_3 found.	Percentage Error.
5	2·5	0·1160	0·1147	1·1
5	2·5	0·1160	0·1156	0·36
5	9·48	0·1160	0·1147	1·1

A comparison of determinations of the concentrations of a fuming nitric acid by direct titration, and by this method, shows that, with these quantities, there is a constant error, probably manipulative, of about 0·7 per cent.; the addition of this loss would not be unjustifiable.

The Stability of Nitric and Nitrous Acids in Acetic Acid Solution.

For testing the stability of nitric and nitrous acids in acetic acid solution, we used the purest glacial acetic acid, which is not attacked by chlorine in the dark (Orton, Edwards and King, *J. Chem. Soc.*, 1911, **99**, 1178).

At first the solutions were carefully preserved in the dark, but, as we found later, exposure to diffused light has no perceptible influence. When nitrous acid is present, the original colour of the solution fades quickly to a scarcely perceptible tint. Concentrations of nitric acid, varying from 1 to 7 per cent., were used. As the nitrous acid disappears, the nitric acid increases somewhat in quantity. No reduction of the nitric acid was observed in any of the solutions which were prepared. The following numbers will illustrate the evidence we obtained:

1. *Glacial Acetic Acid Solutions.*—(*a*) The solution was made up to contain 7·03 grms. HNO_3 and 0·102 grm. HNO_2 per 100 c.c.:

Time after making up.	Grms. of HNO_2 per 100 c.c.	Grms. of HNO_3 per 100 c.c.	
2 hours	0·084	—	—
6 ,,	0·069	—	—
25	0·048	—	—
4 days	0·021	—	—
5 7 hours	—	(i.) 7·198	(ii.) 7·172
7 1 hour	0·0123	(i.) 7·170	(ii.) 7·172
29	0·0026	(i.) 7·247	(ii.) 7·247

(*b*) The solution was made up to contain 1·0164 grms. HNO_3 per 100 c.c.:

Time after making up.	Grms. of HNO_2 per 100 c.c.	Grms. of HNO_3 per 100 c.c.	
7 days	—	(i.) 1·037	(ii.) 1·042

2. *Fifty per Cent. Aqueous Acetic Acid.*—The solution was made up to contain 3·51 grms. HNO_3 and 0·0557 grm. HNO_2 per 100 c.c.:

Time after making up.	Grms. of HNO_2 per 100 c.c.	Grms. of HNO_3 per 100 c.c.	
2 hours	0·044	—	—
45 „	0·015	—	—
96 „	—	(i.) 3·562	(ii.) 3·565

3. *Glacial Acetic Acid Solutions exposed to Light.*—The solution was made up to contain 2·13 grms. HNO_3 and 0·03 grm. HNO_2 per 100 c.c.:

Time of exposure to light.	Grms. of HNO_2 per 100 c.c.	Grms. of HNO_3 per 100 c.c.	
10 hours	0·009	(i.) 2·136	(ii.) 2·128
20 „	0·009	—	—
64 „	—	(i.) 2·167	(ii.) 2·172

The solution was made up to contain 7·02 grms. HNO_3 and 0·100 grm. HNO_2 per 100 c.c.:

Time of exposure to light.	Grms. of HNO_2 per 100 c.c.	Grms of HNO_3 per 100 c.c.	
32 hours	0·052	(i.) 7·11	(ii.) 7·15

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

ANALYSIS OF FOOD AND DRUGS.

Determination of Iron in (Pharmaceutical) Scale Preparations. W. R. Pratt. (*Pharm. J.*, 1912, 88, 641-642.)—For the analysis of *ferri citras cum quinina et strychnia* and other scale preparations of ferric salts, the author prefers the method of the U.S. Pharmacopœia to that of the British. If alkaloids be present they are separated, the residual liquids evaporated until free from ammonia and then diluted. The ferric solution is placed in a stoppered bottle together with hydrochloric acid and potassium iodide, heated in a water-bath, and the iodine liberated is titrated with thiosulphate. The author has investigated the limits of variation of the amount of acid, the time of reaction, and the temperature. He found that the quantity of hydrochloric acid must be at least twice the theoretical quantity, and may be increased to ten times the theoretical quantity without error. If a sufficient quantity of acid be used, the reaction is complete in ten minutes at 40° C., but no appreciable error is introduced by increasing the time to thirty minutes. The most suitable range of temperatures appears to be from 20° to 60° C., but at lower temperatures a longer time is required for the completion of the reaction. J. F. B.

Method of Preparing Lecithin Emulsions and the Subsequent Estimation of their Strength. J. C. Schippers. (*Biochem. Zeitsch.*, 1912, **40**, 189-192.)—The emulsion may be prepared by dissolving a weighed quantity of lecithin in the least possible amount of toluene, and then mixing the solution with a sufficient volume of 0·9 per cent. sodium chloride solution to give the required concentration. After the mixture has been shaken thoroughly, a current of hydrogen is passed through it until all the toluene has been removed, the emulsion being then submitted to centrifugal action, and filtered through cotton-wool. The following method may be employed for checking the strength of the emulsion thus prepared; the method is based on the estimation of the amount of oxygen required to oxidise the lecithin. Ten c.c. of the emulsion are heated to 90° C. for six hours in a stoppered flask with 10 c.c. of a solution consisting of potassium bichromate, 5 grms., 38 per cent. hydrochloric acid, 300 c.c., and water, 700 c.c. After cooling, 10 c.c. of a 5 per cent. potassium iodide solution are added, the mixture is allowed to stand for at least two hours, 30 c.c. of water are then added, and the solution is titrated with $\frac{N}{25}$ thiosulphate solution. A similar titration is made, using 10 c.c. of the sodium chloride employed in making the emulsion, and the difference in the quantity of thiosulphate used in the two titrations is a measure of the lecithin present. Experiments carried out by the author show that 1 c.c. of $\frac{N}{25}$ thiosulphate solution is equivalent to 1·12 mgrm. of lecithin. W. P. S.

Freezing-Point of Milk. J. B. Henderson. (*Aus. Assoc. Adv. Sci.*, 1912, 13.)—Since the reading of a paper before the Australian Association for the Advancement of Science at its meeting in Brisbane (1909), the determination of the freezing-point of all doubtful samples of milk has been continued in the Government Chemical Laboratory, Brisbane.

The added water indicated by the freezing-point is on the average well above that calculated from the 8·5 per cent. solids-not-fat standard, and still further emphasises the fact that the 8·5 per cent. solids-not-fat legal minimum standard is by no means too high.

During the year 1909 every milk sample containing less than 8·5 per cent. of solids-not-fat was found by the determination of the freezing-point to have been watered, and last year the same result was obtained, except in the two following cases:—

	(*a*) August 19, 1910 (4 cows).	(*b*) September 16, 1910 (? Cows).
Total solids	11·62 per cent.	11·6 per cent.
Fat	4·00 "	3·3 "
Solids-not-fat	7·62 "	8·3 "
Ash	0·64 "	0·68 "
Nitrogen	0·487 "	0·474 "
Freezing-point	—0·565°C.	—0·55°C.

But for the determination of the freezing-point there is little doubt that both of these samples would have been adjudged watered. As it was, the freezing-point showed them to be genuine, and the dairymen were warned to raise the quality of the milk to at least the legal minimum by better attention to their herds.

The determination of the freezing-point is useful in distinguishing between naturally poor milks and watered milks, and the author hopes that its use will soon lead to the prosecution of those who add water to a naturally rich milk, but do not add sufficient water to bring the milk below the legal minimum standards. The average composition of the milk of one herd in Brisbane, the result being the mean of 104 samples, is—Fat 4·5 per cent. and solids-not-fat 9·2 per cent. Such a milk could have 7·5 per cent. of water added, and yet pass the ordinary 3 per cent. fat and 8·5 per cent. solids-not-fat standard. The determination of the freezing-point would, of course, at once show the addition of water.

Estimation of Iron in Milk. F. E. Nottbohm and W. Weisswange. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 514-523.)—The small quantities of iron which occur in milk cannot be estimated satisfactorily by means of gravimetric or volumetric methods, and experiments carried out by the author show that the colorimetric method with thiocyanate yields incorrect results owing to the interfering influence of the phosphates and other constituents of the milk ash. It is therefore recommended that the iron be separated by means of "cupferron" (*cf.* ANALYST 1910, **35**, 327; 1911, **36**, 175), and then estimated colorimetrically with thiocyanate. One hundred c.c. of the milk are evaporated, ignited, and the white ash evaporated twice to dryness with hydrochloric acid free from iron. The residue is then dissolved in about 40 c.c. of $\frac{N}{5}$ hydrochloric acid, the solution is transferred to a flask, and oxidised with nitric acid. After cooling, the solution is rinsed into a separating funnel, 2 c.c. of a 5 per cent. "cupferron" solution are added, and, after the lapse of fifteen minutes the mixture is shaken with 25 c.c. of chloroform. The chloroform layer is now separated, evaporated to dryness, and the residue ignited; the iron oxide in this ash is dissolved in hydrochloric acid, the solution is oxidised with nitric acid, then evaporated twice with hydrochloric acid, and the iron estimated colorimetrically as thiocyanate. Experimental proof is given that the method is trustworthy.

W. P. S.

Estimation of Morphine in Opiates. E. Anneler. (*Arch. Pharm.*, 1912, **250**, 186-198.)—Details are given of a critical study of the different methods of estimating morphine in opiates, and especially in "Panopon Roche," a preparation containing hydrochlorides of all the opium alkaloids in a very pure form. It was found that the method of the b.-pt. and its modification in the French Codex was the most generally suitable, but that the results were invariably too low. The error was constant, however, and a correction could be made by adding 30 mgrms. per 50 c.c. of the mother liquor to the amount of morphine found. Of the shaking-out methods the following was the most satisfactory, the average error in the case of "panopon" being 1·5 per cent. too high: From 0·8 to 1·0 grm. of the sample is dissolved in 30 c.c. of water, and shaken with a concentrated solution of 1 grm. of sodium bicarbonate in a little water, which effects the separation of some of the morphine and the bulk of the other alkaloids. The latter are then removed by shaking the liquid with 10 c.c. of chloroform previously saturated with pure morphine (100 c.c. dissolve 0·051 grm. of morphine at 15° C.). After separation of the liquids, the chloroform is drawn off through a small filter, and the residual liquid extracted twice more with 10 c.c. of the saturated chloroform. The filter is then washed with 60 c.c. of a mixture in equal parts of isobutyl alcohol, and chloroform, and the washings transferred to the separating funnel, which is then shaken for about ten minutes and allowed to stand. The layer of isobutyl alcohol, and chloroform (which need not be absolutely clear) is withdrawn, and the extraction of the residue repeated first with 20 c.c. and then with 10 c.c. of the mixed solvents. The extracts are filtered into another separating funnel, where they are shaken with 20 c.c. of $\frac{N}{10}$ hydrochloric acid, and subsequently washed with three successive portions of 10 c.c. of water, after which the filtered acid extract and washings are covered with 30 to 40 c.c. of ether, and the excess of hydrochloric acid titrated, with iodeosin as indicator.

C. A. M.

Estimation of Essential Oil in Mustard. D. Raquet. (*Ann. Chim. anal.*, 1912, **17**, 174-178.)—The usual practice of mixing the mustard with water and allowing it to stand for some time before the essential oil is separated by distillation does not always yield concordant results; in some cases the preliminary digestion requires to be prolonged for an hour or more in order that the whole of the essential oil may be liberated, whilst in others the time required is much shorter. Further, the essential oil is decomposed by micro-organisms, and if the digestion be carried on for too long a time the results obtained will be too low. If, however, dilute alcohol be used in place of water, the digestion may be allowed to proceed for even twenty-four hours without loss of essential oil. Having regard to these conditions, the following method is recommended for the estimation of the oil: Five grms. of the mustard flour are mixed in a 250 c.c. flask with 100 c.c. of water and 20 c.c. of 90 per cent. alcohol; the flask is now closed and set aside for six hours, or heated to a temperature of 35° C. for one hour. The contents are then distilled, and 50 c.c. of the distillate are collected in a 100 c.c. flask in which 10 c.c. of ammonia have been placed previously; 20 c.c. of $\frac{N}{10}$ silver nitrate solution are now added, the distillation is continued until the 100 c.c. flask is filled nearly to mark, and after the flask has been closed with a stopper carrying a long glass tube, the contents are heated to 85° C. for one hour. When cold, the mixture is diluted to 100 c.c., filtered, and the excess of silver is titrated in 50 c.c. of the filtrate by means of $\frac{N}{10}$ thiocyanate solution after the addition of nitric acid. The number of c.c. of $\frac{N}{10}$ silver nitrate used is multiplied by 0·198 to obtain the weight of allyl thiocarbimide in 100 grms. of the mustard. The following percentage quantities of mustard oil (as allyl thiocarbimide) were found in samples of black mustard of different origin: English, 1·39; Greek, 1·20; French, 1·08; Sicilian, 0·99; Italian, 0·99; Bombay, 0·81.

W. P. S.

Detection of Adulterants in Saffron. E. Nockmann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 453-456.)—The presence of added mineral matter in saffron can usually be detected by estimating the amount of ash in the sample, the presence of borax, potassium nitrate, etc., being also ascertained by means of the usual characteristic reactions for these substances. Ammonium salts can also be detected without difficulty in the saffron itself, but the detection of added sugars and glycerol is not such a simple matter, and the author records the results of his experiments with regard to this problem. The following results of analyses of seven samples of saffron of authenticated purity are given:

	In the Original Substance.		In the Dry Substance.			
				Reducing Substances, calculated as Invert Sugar.		
	Water.	Ash.	Aqueous Extract.	Before Inversion.	After Inversion by Official Method.	After Four Hours' Inversion.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Saffron, select ...	8·42	5·01	73·97	23·54	24·05	39·17
„ superior ...	8·63	5·62	71·62	23·60	24·27	39·12
„ Sierra ...	9·23	5·84	70·87	23·45	24·35	38·40
„ Manzanares	8·68	6·83	70·13	22·57	23·35	38·11
„ commercial	13·54	6·05	76·01	23·56	24·44	39·75
„ „	13·49	5·81	74·72	24·35	24·92	39·65
„ „	11·51	6·11	70·83	22·56	24·03	38·84

The inversion process consisted in adding 4 c.c. of hydrochloric acid of sp. gr. 1·19 to 50 c.c. of the aqueous extract, and heating the mixture in a boiling water-bath for four hours, the maximum reduction being obtained after this length of time. The results obtained by the official method of inversion are also given. It will be seen that the presence of added sugar would be indicated plainly by the values for the invert sugar before and after inversion. Glycerol cannot be detected by oxidising the aqueous extract with permanganate and identifying the oxalic acid formed, as genuine saffron yields considerable quantities of oxalic acid when treated in this manner. Attempts to separate added glycerol from saffron by means of the method used in the estimation of glycerol in wine (evaporation with calcium hydroxide and extraction with a mixture of alcohol and ether) did not yield satisfactory results, as saffron itself gave 3 to 4 per cent. of extract which would be counted as glycerol. If, however, the glycerol residue be transferred to a flask, mixed with solid boric acid, distilled until carbonisation is attained, the distillate being collected in water, this distillate may be tested in the following manner: To 5 c.c. of the distillate are added 1 c.c. of albumin solution (1 c.c. of egg-albumin diluted with 7 c.c. of water and filtered through linen), and 18 c.c. of hydrochloric acid containing nitrous acid (0·1 c.c. of 3·6 per cent. potassium nitrite solution to 200 c.c. of hydrochloric acid of sp. gr. 1·18), and the mixture heated in a water-bath to 50° C. The distillate from a sample containing added glycerol yields a green coloration, whilst that from genuine saffron gives a faint red-blue tint. The spectrum of the distillate is also characteristic. When added glycerol is present in the sample, a dark absorption band is seen in the red portion of the spectrum, whilst the distillate from genuine saffron does not exhibit any absorption spectrum.

W. P. S.

Estimation of Nicotine in Tobacco. E. F. Harrison and P. A. W. Self. (*Pharm. J.*, 1912, 88, 718-719.)—In the method recommended, the tobacco is treated with slaked lime and distilled in a current of steam until all the nicotine has passed over into the distillate, as shown by a few drops giving no cloudiness when treated with acid and an excess of iodine. The distillate is received in a measured quantity of standard acid, and at the end of the distillation the distillate is titrated with standard alkali, using litmus as indicator. A further quantity of 10 c.c. of $\frac{N}{1}$ acid is then added to the neutral solution, the latter is evaporated to a volume of 50 c.c., the nicotine is next precipitated by the addition of an excess of iodine, and the ammonia is estimated by distillation. The whole of the volatile alkali is calculated as nicotine in the first instance, and ten times as much iodine as would be required to combine with that amount is dissolved in potassium iodide solution and added to the 50 c.c. of evaporated distillate; after diluting to 100 c.c. the mixture is filtered, 75 c.c. of the filtrate are treated with thiosulphate to destroy the excess of iodine, and then distilled in the presence of a large excess of sodium hydroxide. The quantity of nicotine present is then calculated. It is convenient to work on a weight of tobacco which will yield about 0·5 grm. of nicotine. The authors have proved that nicotine may be separated completely from ammonia by the above-mentioned method, and state that the process as a whole yields accurate results. W. P. S.

Detection of Other Hydrocarbons in Turpentine. Utz. (*Farbenzeit.*, 1912, 17, 1208-1209; through *Chem. Zentralbl.*, 1912, I., 1641.)—Mennechet (*Bull. Commer.*, 1911, No. 9) has proposed a test which consists in shaking 5 c.c. of the turpentine with a small quantity of magenta and 2 drops of nitric acid; at the end of five minutes the mixture remains bright red in the absence of added hydrocarbons, but should 5 per cent. of the latter be present the colour of the mixture is reddish-brown, and light brown or colourless with upwards of 25 per cent. of hydrocarbon. The author has examined this test and finds that, whilst turpentine and its various substitutes and adulterants by themselves give quite characteristic colorations, it is not possible to detect less than 30 per cent. of the substitutes when added to turpentine. The reaction may be used as a rough test, but the colour changes in the case of mixtures are so indefinite that the test cannot be relied on to give useful information as to the presence of adulterants in turpentine. W. P. S.

Critical Remarks on the Direct Estimation of the Total Solids of Wine. C. von der Heide and E. Schwenk. (*Zeitsch. anal. Chem.*, 1912, 51, 429-466.)—For reasons which are given below, the authors consider that the direct method of estimating the total solids of wine (evaporation and drying in the water-oven) should be abandoned, and that the total solids should be calculated from the sp. gr. of the de-alcoholised wine. The accuracy of this sp. gr. may be checked by determining the sp. gr. of the wine and of the alcoholic distillate from the same, Tabarie's formula being used to calculate the sp. gr. of the alcohol-free liquid. The experiments carried out by the authors show that, during the evaporation of the wine and the subsequent drying of the residue, the following changes in the constituents may, and do, take place: (1) Acetic acid and its homologues are not present in the residue; (2) lactic acid is volatilised to a varying extent; (3) tartaric acid is converted partially into *m*. tartaric acid; (4) succinic acid remains unaltered; (5) malic acid is decomposed partially; (6) the non-volatile acids form esters with the glycerol; (7) there is only a slight loss of glycerol; (8) the sugars are decomposed to a very considerable extent. As a result of these changes, the sp. gr. of the aqueous solution of the total solids is less than that of the de-alcoholised wine. The alterations which occur in the proportions of the constituents as a wine ages influence the changes which take place when the wine is evaporated. For instance, the total solids (as estimated directly) of a new wine, which contains much malic acid, but no lactic acid, cannot be compared with the total solids of the same wine after this has been kept for a length of time sufficient for the malic acid to have been converted into lactic acid. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of the Amino-Acids in Plants by Means of the Formaldehyde Method. O. Bailly. (*Bull. Sci. Pharmacol.*, 1912, 18, 702-711; through *Chem. Zentralbl.*, 1912, I, 1640.)—Amino-acids may be titrated in the aqueous extracts of plants by means of the formaldehyde titration method, after the extracts have been treated with barium chloride to remove phosphates, and decolorised by means of quinine silicotungstate. As ammonia is estimated at the same time, this must be estimated separately in another portion of the extract, and an allowance made for its quantity. The details of the process are: One hundred c.c. of the extract are treated with 10 c.c. of $\frac{N}{1}$ hydrochloric acid, 10 c.c. of a 10 per cent. silicotungstic acid solution, and 10 c.c. of a 3·1 per cent. quinine hydrochloride solution, shaken, submitted to centrifugal action, and filtered. The filtrate is shaken with animal charcoal, again filtered, and 78 c.c. of the filtrate are treated with 1 grm. of barium chloride, then rendered alkaline with potassium hydroxide solution, diluted to 100 c.c., and filtered. Twenty-five c.c. of this filtrate (equivalent to 15 c.c. of the original extract) are acidified with $\frac{N}{10}$ hydrochloric acid, 1 drop of a saturated *p*-nitrophenol solution is added, and the solution exactly neutralised with $\frac{N}{10}$ potassium hydroxide solution. Fifteen c.c. of neutral formaldehyde solution are now added, and the amino-acids are titrated with $\frac{N}{10}$ potassium hydroxide solution, using phenolphthaleïn as indicator. Let N denote the number of c.c. required for the titration; the ammonia is then estimated by distilling a further quantity of 50 c.c. of the extract in the presence of magnesia; the quantity of ammonia is expressed in c.c. of $\frac{N}{10}$ acid—let it be n—then the amount of amino-acid in 100 c.c. of the extract expressed in terms of nitrogen, is found by formula: $\frac{(2N-n)\times 0{\cdot}014}{3}$. The results obtained are correct within 3 per cent., except in the case of tyrosine and histidine, when the error may amount to 10 per cent. The following quantities of amino-acids (expressed as nitrogen per 100 grms. of the dry substance) were found in various plants: Turnip leaves, 0·507; tobacco flowers, 0·272; cabbage leaves, 0·886; lucerne, 0·350; carrot tops, 0·280; birch leaves, 0·144; tobacco leaves, 0·420. W. P. S.

Detection and Estimation of Small Quantities of Copper in Plants. E. Guerithault. (*Bull. Sci. Pharmacol.*, 1912, **18**, 633-639; through *Chem. Zentralbl.*, 1912, I., 1639.)—The method employed by the author consisted in dissolving the ash of the plants in hydrochloric acid, separating the silica, precipitating the copper as sulphide, and converting the latter into oxide by ignition; the oxide is then dissolved in nitric acid, and estimated electrolytically and colorimetrically. Peas, beans, maize, rye, wheat, oats, barley, linseed, radish seeds, cress seeds, gentian root, juniper twigs, and verbena wood, were examined, and found to contain from 0·0046 to 0·0171 part of copper per 1,000 parts of dry substance. W. P. S.

Photographic Estimation of the Intensity Distribution in Blood Spectra. W. Heubner and H. Rosenberg. (*Biochem. Zeitsch.*, 1912, **38**, 345-384.)—A photographic method is described for estimating the relative intensity of the absorption of light in the absorption bands of blood spectra, the method depending on the microphotometric measurement of the light which is transmitted by the negative on which the absorption spectrum has been photographed. Under definite conditions, the extinction of light by the precipitated silver is a measure of the light absorbed by the blood colouring matters. Blood from rabbits, sheep, and pigs, was examined, and showed no difference in the intensity distribution. W. P. S.

Rate of Evolution of Hydrocyanic Acid from Linseed under Digestive Conditions. S. H. Collins. (*Univ. Durham Phil. Soc. Proc.*, 1912, **4**, 99-106.)—The author has digested linseed at temperatures approaching those of animal life, and has removed hydrocyanic acid by a stream of inert gas (hydrogen, air, etc.). The inert gas, passing at a regular rate obtained by means of a gas-governor (ANALYST, p. 330), bubbles through a vessel immersed in a thermostat (*loc. cit.*), the vessel containing the linseed to be tested and 50 c.c. of water. The vapours are caught in an absorbing vessel containing 5 c.c. of $\frac{N}{30}$ sodium hydroxide. A clock and meter are attached to the apparatus immediately before the issuing of the gas, and thus the rate of flow, the time, and the temperature are controlled. Generally, 10 litres of gas per hour at 45° C. were the conditions employed. From time to time the sodium hydroxide in the absorbing vessel is removed by a tap, and fresh quantities of alkali are added through an attached tap-funnel, so that several fractions of 5 c.c. of the hydrocyanic acid-containing solution are obtained. To each of these is added 1 drop of a solution of ferrous sulphate (12 per cent.), the liquids shaken up and allowed to stand for an hour, when 1 drop of diluted sulphuric acid (1 : 2) is added, and, after shaking, the liquids allowed to stand at least thirty-six hours. The colours are matched after three, four, and five days with standards, made from potassium cyanide solution of known strength, prepared with iron and acid similarly. Standards of prussian blue keep well (twelve months), 0·01 mgrm. to 0·30 mgrm. HCN being useful quantities to prepare. The mean reading of the separate days is taken as correct.

The amount of hydrocyanic acid yielded by linseed, and the rate of its yield, depend upon the amount of cyanogenetic glucoside and enzyme, on the temperature, degree of acidity of the liquid present, and on the presence or absence of a number of other substances. In normal health the acidity of the stomach is too high to allow of evolution of hydrocyanic acid, but under abnormal conditions—for example, in the case of a linseed rich in cyanogenetic glucosides fed to a beast suffering from indigestion such that the food was not rendered acid—hydrocyanic acid poisoning may set in. Acids of $\frac{N}{100}$ strength prevent the action of the enzyme, and hydrochloric acid of $\frac{N}{1000}$ retards the evolution of hydrocyanic acid. Probably all acids prevent this action.

The following are some of the results obtained by the above method:

Linseed (Calcutta).			Linseed Cake.	
Minutes.	Sample 1. HCN mgrm.	Sample 2. HCN mgrm.	Minutes.	HCN mgrm.
26	0·103	0·100	23	0·000
61	0·243	0·243	59	0·057
122	0·333	0·336	125	0·210
195	0·370	0·372	314	0·393
305	0·374	0·382	366	0·446
	(=98·5 %)	(=100·5 %)		(=100 %)

Linseed (Calcutta) and 50 c.c. of $\frac{N}{200}$ HCl.		Linseed (Calcutta) and 50 c.c. $\frac{N}{100}$ Lactic Acid.	
Minutes.	HCN mgrm.	Minutes.	HCN mgrm.
24	0·000	23	0·000
59	0·024	51	0·000
130	0·104	125	0·022
225	0·191	216	0·055
325	0·272	300	0·086
	(=71·6 %)		(22·6 %)

A. R. T.

Quantitative Estimation of *l*-β-Hydroxybutyric Acid in Urine and Blood.

B. O. Pribram. (*Zeits. exper. Path. u. Ther.*, 1912, **10**, 279-283; *Chem. Zentralbl.*, 1912, I., 1509.)—The method is based on the fact that the crotonic acid produced by the distillation of β-hydroxybutyric acid in sulphuric acid solution combines quantitatively with two atoms of bromine. The conversion of the β-hydroxybutyric acid into the volatile crotonic acid is best effected by means of sulphuric acid of 58 to 60 per cent. by weight. An excess of standardised bromine water is added to the distillate and titrated back with thiosulphate. In the estimation of β-hydroxybutyric acid in diabetic urine, the method is complicated owing to the presence of phenols and sugar. The difficulty is surmounted by saturating the urine with ammonium sulphate, acidifying with sulphuric acid, and extracting with ether. The estimation is made on the ethereal extract. Blood is defibrinated before the estimation by a preliminary treatment with six times its quantity of alcohol. J. F. B.

Use of Mice and Birds for Detecting Carbon Monoxide after Mine Fires and Explosions. G. A. Burrell. (Technical Paper II., Bureau of Mines, Washington, 1912.)—The formation of a cap on the safety-lamp flame is not satisfactory as a means of detecting carbon monoxide. Blood solution and cuprous chloride solution show its presence with considerable sensitiveness, but the best method is the use of mice or birds. When exposed to an atmosphere of carbon monoxide, canaries show signs of poisoning sooner than mice, and the symptoms are more easily observed. The author remained for twenty minutes in an atmosphere containing 0·25 per cent. of carbon monoxide, without experiencing more than a slight headache, though later he became ill for several hours; canaries in the same atmosphere showed distress in one minute and fell from their perches in five. A party exploring a mine in which an explosion had taken place, carried with them a canary, which collapsed on reaching a spot at which there was 0·2 to 0·3 per cent. of carbon monoxide; the party retreated without distress. O. E. M.

Modification of Wohlgemuth's Method for the Quantitative Investigation of the Pancreas Function. P. B. Hawk. (*Arch. Internal Med.*, 1911, **8**, 552-556, through *Chem. Zentralbl.*, 1912, I., 1477.)—Wohlgemuth (*Berl. Klin. Wochensch.*, 1910, **47**, 92) described a method for the quantitative estimation of fæcal-amylase. According to this method, a weighed quantity of fæces is extracted with a 1 per cent. sodium chloride solution, the extract is centrifuged, and the amylolytic power of regularly decreasing quantities of this extract is determined at 38° C. by the hydrolysis of 5 c.c. of a 1 per cent. solution of soluble starch. After twenty-four hours' action, iodine solution is added, and the degree of digestion is estimated colorimetrically. The author's experiments have shown that this method gives satisfactory results in the case of fæces having a reaction within the normal limits, but in the case of strongly acid fæces false results may be obtained, owing to the extreme sensitiveness of the amylase towards acids. In order to avoid this source of error, the author recommends, for the extraction of the fæces, a solution of 0·1 mol. of sodium dihydrogen phosphate and 0·2 mol. of disodium hydrogen phosphate in 1 litre of 1 per cent. sodium chloride solution. J. F. B.

Separation of Peroxydase from Catalase. A. Kasanki. (*Biochem. Zeitsch.*, 1912, **39**, 64-72.)—The method described is based upon the fact that catalase is precipitated when treated with pyrogallol, whilst peroxydase remains in solution.

On adding pyrogallol to the clear filtered juice obtained from hemp seedlings in quantity sufficient to make the concentration of the precipitant between 4 and 5 per cent., a precipitate is formed which is free from peroxydase; the latter, however, passes into the filtrate when the mixture is filtered. If the pyrogallol be added in sufficient quantity, the catalase is rendered inactive, and cannot be detected in either the precipitate or the filtrate. The method may be employed for preparing catalase-free peroxydase from different sources. W. P. S.

Experiments with the Castor-Bean Lipase. K. G. Falk and J. M. Nelson. (*J. Amer. Chem. Soc.*, 1912, **34**, 735-745.)—A comparative study was made of the hydrolysis of methyl acetate, ethyl butyrate, and olive oil, caused by the castor-bean lipase. No appreciable influence on the subsequent hydrolysis was found when small amounts of alkali were added at the beginning of the action on methyl acetate, but with ethyl butyrate the action was different, depending on the amounts of alkali added. Corrections were introduced in every case for the acid formed from ester solvent and lipase solvent. When the solvents used were ether saturated with water, and acetone containing a small amount of water, methyl acetate was hydrolysed to a considerable extent with lipase, both at the ordinary and at more elevated temperatures. Small amounts of an active constituent were extracted from the lipase preparation by water and by ethyl acetate. By the electrolysis in water of the lipase preparation a substance was produced in the anode solution, probably by oxidation, showing marked hydrolytic action. In all cases the amount of ester hydrolysed was measured by titrating with a $\frac{N}{10}$ aqueous solution of sodium hydroxide, using phenolphthaleïn as indicator. A large number of experiments are described with details as to temperature, time of action, solvent used, and titration figures. The lipase preparation used was prepared by grinding the castor-beans roughly, extracting a number of times with carbon tetrachloride or chloroform, and then grinding to a fine powder and passing through a hundred-mesh sieve and thus separating the majority of the shells. The fine powder was then exhaustively extracted with ether, leaving a light grey preparation containing 9 per cent. of ash, which, when mixed with water, had always a slightly acid reaction, increasing slowly on standing.

H. F. E. H.

Biochemical Method for the Estimation of Small Quantities of Salicylic Acid in Presence of Excess of p-Hydroxybenzoic Acid. J. Böeseken and H. Waterman. *K. Akad. van Wetensch., Amsterdam*, 1912, **20**, 548-552; through *Chem., Zentralbl.*, 1912, I., 1503-1504.)—*p*-Hydroxybenzoic acid is capable of being utilised by *Penicillium glaucum* as a source of carbon nutrition, whereas salicylic acid exerts a retarding influence on the growth of this organism. For the estimation of small quantities of salicylic acid in the presence of *p*-hydroxybenzoic acid, the following process may be adopted: 3 grms. of *p*-hydroxybenzoic acid, 0·5 grm. of dipotassium hydrogen phosphate, 0·5 grm. of ammonium chloride, and 0·2 grm. of magnesium sulphate, are dissolved in 1 litre of water, and several Erlenmeyer flasks are charged with equal quantities of the solution; increasing quantities of salicylic acid are added to the series of flasks, the contents are sterilised, and each flask is infected with a culture of *Penicillium.* A 0·3 per cent. solution of the acid mixture to be analysed is treated in the same manner, and the development of mould-fungus growth which takes place in the latter is compared with that in the flasks containing the standard mixtures. J. F. B.

Refractometer Value of the Serum of Cow's and Goat's Milk. K. Alpers. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 497-513.)—The author considers that the refractometer value of the calcium chloride serum of milk, as determined by the process described by Ackermann (Analyst, 1907, **32**, 117) is of value as a criterion in the detection of added water in milk. For the milk of separate healthy cows the value lies between 35·8 and 41·3 at 17·5° C., the average value for 126 samples of milk examined being 39·11. The daily variation observed did not exceed 1·4. Goat's milk showed values of from 34·7 to 40·3, with an average of 38·0, a value slightly lower than that given by cow's milk. W. P. S.

Value of Direct Enumeration of Bacteria in Water by Means of the Ultra-Microscope. Aumann. (*Zentralbl. Bakt.-u.-Parasitenk.*, 1912, II., 624-635; through *Chem. Zentralbl.*, 1912, I., 1594.)—The ordinary method of counting bacteria in the glass apparatus of Thomas and Zeiss is regarded as unsuitable, owing to the difficulty of completely sterilising the glass, though this objection does not apply to apparatus of quartz. The use of the latter is as a rule only practicable with water containing many bacteria, as, for example, in detecting large leakages in filtration. For the general examination of water the counting method with the ultra-microscope does not afford any certain conclusion as to the number of micro-organisms present, much less as to the suitability of the water for drinking purposes. C. A. M.

ORGANIC ANALYSIS.

Methods of Analysing Coal and Coke. F. M. Stanton and A. C. Fieldner. (U. S. Dept. of Interior, Bureau of Mines, Technical Paper, 8, pp. 19.)—This publication describes in detail the methods of analysis in use in the laboratories of the Bureau of Mines, and is published in consequence of the numerous requests addressed to the Bureau for information as to its methods. The methods are substantially those recommended by the American Chemical Society, but a few modifications have been introduced, and somewhat elaborate apparatus is figured. The practice in the Bureau laboratories is to dry the whole 3-pound samples received by them to constant weight at 30°-35° C., and an oven specially designed for this service is figured. After quartering down to about 400 grms., the sample is then reduced to 60 mesh in a ball mill. Moisture is determined in the usual way by drying for one hour at 105° C., and a glycerol oven of quite novel form, which is said to insure great uniformity of temperature, is illustrated. The greatest deviation from the methods of the American Chemical Society is in the use of a sheet-iron chimney to protect the crucible from draughts during the determination of volatile matter. The publication also includes descriptions of apparatus for the ultimate analysis of coal, and directions for the use of the bomb calorimeter and for the determination of true as well as apparent specific gravity. G. C. J.

Volatile Acidity of Gum Tragacanth compared with that of Indian Gum. W. O. Emery. (*J. Ind. and Eng. Chem.*, 1912, 4, 374-376.)—The author has investigated the question of the detection of Indian gum in admixture with tragacanth by a method based on the estimation of the volatile acid produced on hydrolysis with acids. Indian gum is derived from the *Sterculia urens* or the *Cochlospermum gossypium*, the specimens examined belonging to the former species. The quantitative estimation of the volatile acidity is carried out in the following manner: One grm. of the whole or powdered gum is treated in a 700 c.c. round-bottomed flask, provided with a long neck, for several hours in the cold with 100 c.c. of water and 5 c.c. of syrupy phosphoric acid until the gum is completely swollen. The mixture is then boiled gently for two hours under a reflex condenser, whereby a clear colourless solution is obtained, a very small amount of cellulosic residue remaining. The hydrolysed product is next distilled slowly in a vigorous current of steam until the distillate amounts to 600 c.c. and the acid residue to about 20 c.c., care being taken not to scorch the organic matter in the flask. The spraying of phosphoric acid may be prevented by fitting a spray-trap to the still-head. The distillate is titrated with $\frac{N}{10}$ potassium hydroxide in presence of 10 drops of phenolphthaleïn solution, finally boiling the liquid under titration until a faint pink colour persists. The result is controlled by a blank distillation with the omission of the gum. The volatile acidity is expressed in terms of c.c. of $\frac{N}{10}$ alkali per 1 grm. of gum. A number of authentic samples of gum tragacanth showed, for lumps, 3·2 to 4·2 c.c., mean 3·6 c.c.; for powder, 3·5 to 4·1 c.c., mean 3·7 c.c., equivalent to 2·2 per cent. of acetic acid from the original gum. As regards Indian gum, it has previously been shown by Robinson that the gum of *Cochlospermum gossypium* yields on hydrolysis 14·4 per cent. of acetic acid. The author has treated several samples of the gum of *Sterculia urens*, and found—for lumps, 25·9 to 28·3 c.c., mean 26·7 c.c., equivalent to 15·9 per cent. of acetic acid; for powder, 25·4 to 27·7 c.c., mean 26·5 c.c., equivalent to 15·89 per cent. of acetic acid. It is noted that, whereas gum tragacanth yields practically a colourless solution after hydrolysis, the gum of *Sterculia urens* yields a rose-pink solution on boiling with phosphoric acid. It is concluded that the volatile acidity values are sufficiently constant to afford a good means of detecting and estimating admixtures.

J. F. B.

Investigations on Mercury Fulminate. R. Philip. (*Zeitsch. ges. Schiess. u. Sprengstoffwesen*, 1912, 7, 109, 156, 180, 198, 221.)—The Brownsdon method for the estimation of fulminate (*J. Soc. Chem. Ind.*, 1884, 3, 17; 1905, 24, 382) consists in determining, without delay, the alkalinity produced by excess of sodium thiosulphate on the fulminate, using methyl orange. This was modified to eliminate the error due to the reduction of the alkalinity on standing, by using a large excess of thiosulphate, and working always under standard conditions. A method in which the excess of thiosulphate is titrated back with iodine was also evolved, and finally, a combination was found to give good results. Three grms. of pure potassium iodide (the use of which reduces the loss of alkalinity) are dissolved in 50 c.c. $\frac{N}{10}$ thiosulphate solution, 0·3 grm. of fulminate is washed in and dissolved by agitation with a rotary motion, and the solution titrated first with methyl orange and $\frac{N}{10}$ acid, then with starch and $\frac{N}{10}$ iodine (1 c.c. $\frac{N}{10}$ acid = 0·0071 grm. mercury fulminate; 1 c.c. $\frac{N}{10}$ iodine = 0·0142 grm. mercury fulminate). In most cases the results were consistent, and the two titrations agreed satisfactorily, the iodimetric ones being somewhat the higher; but with certain fulminates purified by crystallisation from potassium cyanide, the alkalimetric titration apparently showed 1·2 per cent. more mercury fulminate than the iodimetric. This is attributed to the possible presence of a product of the partial decomposition of the fulminate by the solvent. It was found that brown fulminate is not necessarily less pure than white. O. E. M.

Analysis of Indigos containing Starch. G. H. Frank and A. G. Perkin. (*J. Soc. Chem. Ind.*, 1912, **31**, 372-373.)—It has been shown by Thomson (ANALYST, 1911, **36**, 290) that the sulphonation methods of Rawson or Bloxam do not give accurate results in the analysis of indigo containing starch. The author's experiments upon mixtures of pure indigotin and starch confirm Thomson's conclusions, and show that the low figures obtained are probably due to the destruction of indigotin through a reducing action of the products of hydrolysis of the starch during the sulphonation. This process is counterbalanced more or less by an oxidising process, when fuming sulphuric acid is used for the sulphonation, as in Bloxam's process; and this would explain the concordance of the results obtained by Bergtheil and Briggs (*J. Soc. Chem. Ind.*, 1906, **25**, 733) with mixtures of indigotin and starch. Further experiments have shown that pure indigotin is not altered when digested with dilute hydrochloric acid in the presence of starch, and that there is no reason against the method of extracting the starch from a mixture by treatment with boiling dilute hydrochloric acid, and estimating the indigotin in the residue by the tetrasulphonate process. Only by boiling the mixture for a long time with the potassium acetate were the results too low with Bloxam's process; but this is unnecessary, and a temperature of about 90° C. is quite sufficient for obtaining a clear solution. C. A. M.

Occurrence of Methylcyclopentenolon in Pyroligneous Acid. J. Meyerfeld. (*Chem. Zeit.*, 1912, **36**, 549-552.)—A hitherto unknown unsaturated ketonic alcohol of the pentamethylene series, methylcyclopentenolon, $C_6H_8O_2$, has been isolated by the author from pyroligneous acid. In the pure condition it melted at 106° C., and sublimed with slight decomposition at 210° C. It had a characteristic odour and a sweet pungent taste. It could readily be obtained in crystalline needles from hot water, alcohol, acetone carbon tetrachloride, etc. In aqueous solution it gave a deep violet stable coloration with ferric chloride. It reduced ammoniacal silver nitrate on gently heating, and Fehling's solution on boiling, and with phenylhydrazine yielded an osazone, $C_{18}H_{18}N_4$, which crystallised from alcohol in yellow needles, melting at about 140° C. It yielded salts with metals, and from these the ketonic alcohol could be regenerated by treatment with a mineral acid.

C. A. M.

New Reaction for Organic Bases. Charitschkoff. (*Chem. Zeit.*, 1912, **36**, 581.)—A solution of the so-called inactive naphthenic acid in hydrocarbons or in ether does not react with copper sulphate by itself; but as soon as a trace of aniline, pyridine, quinoline, piperidine, coniine, codeine, nicotine, quinine, or other base that is soluble in ether is added, the reaction takes place with the formation of copper naphthenate. On this reaction is based a sensitive test for the detection of the above-named basic compounds, a solution of the inactive naphthenic acid in ether or petroleum spirit, with the addition of 3 per cent. of copper sulphate solution being used as the reagent. On shaking the mixture the liquid remains colourless until a trace of a base is added. It is suggested that the test may be of value for the detection of alkaloids and ptomaines in physiological investigations. Caffeine and diphenylamine do not give the reaction. Oleic acid may take the place of inactive naphthenic acid in the test. In the case of crystalline bases and alkaloids which are only sparingly soluble in ether or petroleum-spirit, free acid or amyl alcohol may be used for the extraction. C. A. M.

Use of Phenolphthaleïn as an Indicator. J. W. McBain. (*J. Chem. Soc.*, 1912, **101**, 814-820.)—Stress is laid upon the great influence of extraneous carbon dioxide upon the results of a titration, and it is shown that in the presence of that gas in solution about ten minutes are required for the final end-point of the reaction to be reached. In the absence of carbonate, however, or if much carbonate is present, as in the case of a normal bicarbonate solution, the end-point occurs practically instantaneously and is permanent. The effect of carbon dioxide (carbonates) upon the sensitiveness of the end-point was estimated by titrating solutions of potassium hydroxide freed from carbon dioxide and carbonate, with and without the addition of known quantities of carbonate. It was found that with the greatest care the amount of alkali needed to change the colour was about fifty times greater than the 0·0043 c.c. of $\frac{N}{10}$ alkali anticipated. The changes of colour in these experiments were compared with those of a colour scale ranging from No. 1 to 200, the first solution matching in colour that of a solution of 0·50 grm. of cobalt nitrate and 0·62 grm. of copper sulphate per litre, and the others containing two, three, four, etc., times these amounts. For comparing the colours above No. 10, a Donnan's colorimeter was used, and the colour of a solution nearly saturated with sodium phosphates and coloured with phenolphthaleïn was standardised against the cobalt and copper solutions. The difference between each member of the standard series was intended to correspond to the change in colour produced by the addition of one drop of $\frac{N}{250}$ acid or alkali. McCoy (*Amer. Chem. J.*, 1903, **29**, 437) gives the alkalinity of $\frac{N}{10}$ sodium hydrogen carbonate as $2{\cdot}9 \times 10^{-6}N - OH'$, which would correspond to about No. 70 in the phenolphthaleïn standard. Hence, the amount of alkali required to produce the colour change in a titration, considered in conjunction with McCoy's value, affords a sensitive means of estimating the amount of carbonate in a given solution. Estimations made with the different strengths of phenolphthaleïn solution showed that a pale colour, corresponding to about No. 3 in the standard scale, was the most sensitive for the end-point, and was also closer to the point of theoretical neutrality. It is advisable always to use about 20 drops of a 1 per cent. alcoholic solution of the phenolphthaleïn per 100 c.c.; since when carbonate is present the depth of colour is approximately proportional to the amount of phenolphthaleïn added up to the saturation point (50 to 60 drops). The error caused by adding only 1 drop instead of 20 drops of the indicator to 100 c.c. of distilled water corresponded to 0·08 c.c. of $\frac{N}{10}$ potassium hydroxide solution. The phenolphthaleïn solution had been freed from carbon dioxide, neutralised with alkali, and was protected by a guard-tube of potassium hydroxide. With regard to the degree of accuracy possible in ordinary titrations, 0·01 per cent. was found to be the limit of agreement in duplicate estimations when large quantities of acid and alkali were used, and all precautions were taken in the ordinary atmosphere. As a precaution it is suggested that the water in the titration flask should be coloured to a pale tint similar to that to be attained at the end-point, before adding the acid, and that care should be taken not to breathe in the direction of the flask. Under these conditions the results of two estimations should agree within 0·1 per cent. C. A. M.

Simultaneous Effect of Alcohol and a Neutral Salt on the Sensitiveness of Phenolphthaleïn. E. Lenk and G. Mondschein. (*Chem. Zeit.*, 1912, **36**, 534.)—The effect of varying amounts of water on the system-ammonium chloride, water, phenolphthaleïn—and of water and alcohol on the system—ammonium chloride-water-alcohol-phenolphthaleïn was investigated. Alcohol displaces the end-point very much more than does water. For example, 10 c.c. of 2 N ammonium chloride solution, neutral to litmus, required 2·4 c.c. $\frac{N}{10}$ potassium hydroxide solution to give a coloration with phenolphthaleïn; the addition of 20 c.c. of neutral water increased this to 3·8 c.c., while 20 c.c. of alcohol raised it to 38·0 c.c. This is not an ionisation phenomenon, but is due to constitutional alterations in the indicator.

O. E. M.

INORGANIC ANALYSIS.

Use of Arsenious Acid in Volumetric Analysis. II. Estimation of Mercury. F. Litterscheid. (*Chem. Zeit.*, 1912, **36**, 601.)—The solution of mercuric salt, which should not contain less than 0·1 grm. nor much more than 0·4 grm. of mercury, and which should have a volume of about 50 c.c., is mixed with 50 c.c. of $\frac{N}{10}$ arsenious acid solution and 5 grm. sodium bicarbonate in a 200 c.c. flask, and the mixture is heated on the water-bath until the precipitate acquires the dark grey tint of finely-divided metallic mercury. If the flask be shaken from time to time, the reaction is complete in half an hour. The contents of the flask are then diluted somewhat, cooled, and finally made up to 200 c.c. Since the mercury is very finely divided, the mixture is filtered through a double hard filter, or a teaspoonful of precipitated chalk is mixed with the contents of the flask before filtering through an ordinary paper. The first runnings are rejected, and the subsequent ones returned to the filter until 100 c.c. of clear filtrate has been collected. This is then diluted to about 500 c.c., and, after addition of a further 5 grms. of sodium bicarbonate, the excess of arsenious acid is determined by means of $\frac{N}{10}$ iodine solution. If the original solution contained ammonium salts, the filtrate from the reduced mercury is first acidified with sulphuric acid and then saturated with sodium bicarbonate (10 grms.). G. C. J.

System of Qualitative Analysis for the Common Elements. Part V. Acidic Constituents. A. A. Noyes. (*J. Amer. Chem. Soc.*, 1912, **34**, 609-643.)—This paper forms a continuation to those previously published by the author under the same main title (*J. Amer. Chem. Soc.*, 1927, **29**, 137; 1908, **30**, 481; 1909, **31**, 611). In the aggregate, these papers cover more than 200 pages, and constitute in effect a textbook of qualitative analysis, largely based on the author's original investigations, but necessarily embodying so much that is common knowledge that no ordinary abstract of them is possible. The scope of the present paper is indicated in its sub-title, and the system of analysis is based on the volatilisation of most acids by boiling with phosphoric acid, collecting the distillate in two portions, the first of which contains the readily volatile, slightly ionised acids, the second the less volatile, largely-ionised acids. In the search for individual acids, the attempt has been made to select or devise tests of known and roughly equal sensitiveness, and preference has been given to those methods which enable the experienced analyst to form an approximate estimate of the quantities present. G. C. J.

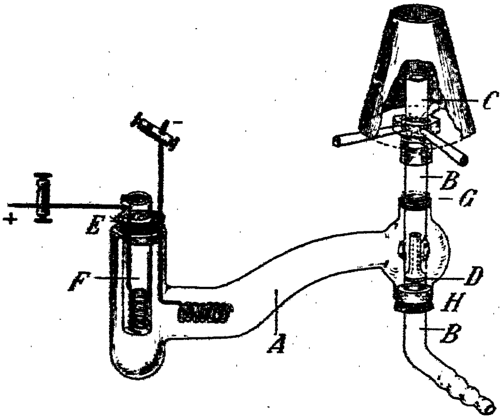
Study of Cobaltinitrites and their Application to Analytical Chemistry. L. L. Burgess and O. Kamm. (*J. Amer. Chem. Soc.*, 1912, **34**, 652-659.)—The sensitiveness of the cobaltinitrite test for potassium may be greatly increased by having silver nitrate present, the double cobaltinitrites of potassium and silver—$Ag_2KCo(NO_2)_6$ and $AgK_2Co(NO_2)_6$—being much less soluble than potassium cobaltinitrite or sodium di-potassium cobaltinitrite, one or other of which, according to the amount of reagent used, constitutes the precipitate of the ordinary test. For example, when 1 drop of a freshly prepared 25 per cent. solution of sodium cobaltinitrite is added to a solution containing less than 100 parts per million of potassium, no perceptible precipitate is formed. If the test is repeated in presence of $\frac{N}{100}$ silver nitrate, a copious yellow precipitate is formed immediately. In presence of $\frac{N}{100}$ silver nitrate, solutions containing only 2 parts of potassium per million give an almost immediate precipitate, which persists for half an hour, and a solution containing only 0·5 part per million gives a distinct precipitate within fifteen seconds, the precipitate persisting for about five minutes. In applying this test, it is essential that the solution be neutral, or only slightly acidified with acetic acid. Halogens are conveniently removed by addition of silver nitrate and filtering, such an excess of silver being used as will make the filtrate an approximately $\frac{N}{100}$ solution of silver nitrate. In the usual scheme of analysis, the test may be applied after removal of the heavy metals. The filtrate from the latter is acidified with an excess of nitric acid, evaporated to dryness and ammonium salts volatilised, since ammonium silver cobaltinitrite is so insoluble that the test here described would serve for the detection of ammonia in concentrations of 5 parts per million in absence of potassium. Rubidium, caesium, and thallium, form double cobaltinitrites with silver which are even less soluble than the corresponding potassium salt. No other metal is likely to interfere, for, though barium forms sparingly soluble cobaltinitrites, it will not interfere, unless the concentration of barium exceeds $\frac{N}{10}$ strength. G. C. J.

Separation of Iron from Titanium. F. Bourion. (*Comptes rend.*, 1912, **154**, 1229-1232.)—The substitution of sulphur monochloride for chlorine in the process where iron is separated from titanium by volatilisation in a current of chlorine and hydrogen chloride simplifies the apparatus necessary for this separation, and permits of its being accomplished at a lower temperature. To prevent volatilisation of titanium, it is necessary to keep the concentration of sulphur monochloride low, but this is readily accomplished as follows: The sulphur monochloride (2 c.c.) is contained in a flask of 160 to 170 c.c. content, surrounded by melting ice, and the stream of dry hydrogen chloride is introduced into the flask by a tube which comes within 4 to 10 mm. of the surface of the liquid, the mixed gases then passing to the combustion tube in which the mixed oxides of titanium and iron are contained in a porcelain boat. At its forward end the combustion tube is connected to a Will and Varentrapp apparatus, charged with water. The reaction commences at 200° C., and until a temperature of 400° C. is reached the gas stream may be as rapid as seventy-five bubbles per minute, but should then be reduced to half this rate, whilst the temperature is raised to 700° to 750° C., at which the reaction is complete in from two and a half to four hours with 0·3 to 0·5 grm. substance. After allowing the tube to cool, the hydrogen chloride in it is expelled by means of a current of dry air. Under the conditions described, no trace of iron will be left in the boat, nor can titanium be detected, even by the peroxide test, either in the contents of the receiver or in the washings of the combustion tube. In these washings and the contents of the receiver the iron is precipitated by means of ammonia after oxidation of any ferrous iron by means of nitric acid. Occasionally magnetic oxide of iron may be formed in the forward part of the tube, as the result of the action of the steam of reaction on ferrous chloride, and the analyst must bear this in mind and take appropriate steps for bringing this iron into solution. G. C. J.

Estimation of Radium in Minerals and Rocks. E. Ebler. (*Chem. Zeit.*, 1912, **36**, 604.)—The method depends on the determination of the quantity of emanation given off in some definite interval of time by a known weight of the mineral. The weighed substance is placed in a platinum boat, either alone or mixed with some suitable flux, such as potassium carbonate, borax, or alkali phosphates (*Cf.* Joly, *Phil. Mag.*, 1911 [vi], **22**, 134; Eve and McIntosh, *Trans. Roy. Soc. Canada*, 1910 [iii], **4**, III., 67), and is heated to free it completely from emanation. The boat is then placed in a quartz combustion tube, which lies in an electric furnace, and which is connected by ground connections and taps to a mercury pump. After an interval of time, which may vary from two days to a month, according to the amount of radium present, the gaseous contents of the tube are transferred by means of the pump to a small gasholder, the contents of the boat being strongly heated towards the close of the operation. The air and emanation are then transferred to an evacuated ionisation chamber, and the radium emanation measured in the usual manner by determination of the saturation current. The absolute weight of radium is arrived at by means of a control experiment on a weighed quantity of some substance of known radium content. For use as a standard, radium barium oxalate of approximately 10 per cent. radium content serves well, and its real radium content may be estimated with an accuracy of 0·5 per cent. by determining its mean molecular weight, for which purpose a small quantity is weighed on a micro-balance, converted into oxide, and reweighed. The use as standards of mixtures of radium and barium salts of organic acids of higher molecular weight than oxalic acid would increase the accuracy of the method, and experiments with this object in view are in progress. The method, as described, permits of the estimation of radium in minerals with an accuracy of about 0·5 per cent.

G. C. J.

Spectroscopic Process for the Estimation of Small Quantities of Rubidium in Presence of much Potassium. E. Wilke-Dörfurt. (*Zeitsch. anorg. Chem.*, 1912, 75, 132-140.)—The process consists in dissolving the substance (potassium salts) in water, feeding atomised spray from this solution to a special burner, photographing a portion of the spectrum and comparing the photograph so obtained with a series of standards prepared under nearly identical conditions. The illustration shows the atomiser and burner. The glass burner tube *B* is of 9 mm. internal diameter and extends for 7 cm. above the point *D*, where a capillary, 3 cm. long and 0·8 mm. in internal diameter, is fused into it. Opposite the capillary jet are two openings for the supply of air to the burner through the atomising device *A*. To the top of the glass burner tube, a porcelain tube of the same internal diameter and 5 cm. long is attached by means of rubber tubing. Rubber tube is also used for making the joints at *G* and *H*, where the burner tube enters and leaves the bulb-shaped extension of the vessel *A*. The vessel *A* is constructed from tubing of 2·8 cm. internal diameter, the height of the anode chamber being 8 cm., and the length of the knee-shaped portion, the lower part of which accommodates the cathode, 12 cm. The bulb through which the burner tube passes is of 3·5 cm. diameter, and the distance between *G* and *H* 5·5 cm. *E* is a cork sufficiently nicked to allow a free supply of air to the burner. The cathode wire passes between the cork and the glass wall of the tube, but the anode is suspended within a short length of glass tubing, which serves to allow the escape of electrolytic oxygen and traces of

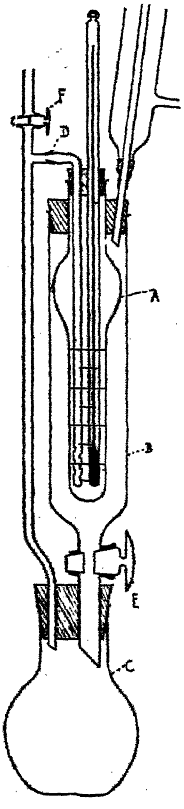


chlorine. The colouring of the flame is effected, on the Beckmann-Riesenfeld principle, by electrolytic atomising of the solution, of which 40 c.c. is placed in the vessel *A*. The solution should be a 10 per cent. solution of the mixed salt, as a high concentration of potassium insures the current being conducted by the potassium chloride, and avoids any possibility of rubidium becoming transformed into hydroxide or perchlorate. The electrodes are of 0·8 mm. platinum wire and a current of 2·5 ampères at 4·5 volts gives a suitable evolution of gas. For photographic work, the most suitable portion of the spectrum to select is that between 396 $\mu\mu$ and 435 $\mu\mu$, that is to say the blue, not the red, which serves best for work with the naked eye. The spectroscope used by the author has a prism, the dispersion of which between *c* and *h* amounts to 4·25°; the opening of the collimator and of the camera is 19 mm. As the inner cone of the Bunsen flame gives lines in the blue, the inner cone of the flame is kept as small and sharply defined as possible. Obviously, in attempting to make a process such as this quantitative, every precaution must be taken to work under constant conditions. Among important conditions, easily controlled, are gas pressure, region of flame observed, time of exposure, and manner of development of the plates. Uniformity in manufacture and age of the plates is insured by buying direct from the makers. The author uses Agfa "extra rapid" plates, and gives a half-hour exposure. This length of exposure tends to eliminate errors due to small periodical changes in some of the conditions which are difficult to suppress, but which neutralise each other during a prolonged exposure. Too long an exposure, however, leads to the appearance of a continuous spectrum, due to dust particles, which reach the flame, in spite of all precautions. The author describes the development of his plates, but any process, provided it be a rigidly uniform one, may be used. For concentrations of rubidium relative to potassium of from 0·05 to 10 per cent., the method is accurate to within about 10 per cent. It was designed to estimate only the order of magnitude of the rubidium percentage in new potash workings, but has proved more accurate than this, as stated. G. C. J.

Estimation of Sulphides in Lime Liquors. J. R. Blockley and P. V. Mehd. (*J. Soc. Chem. Ind.*, 1912, **31**, 369-372.)—Estimation of the sulphides in lime liquors for leather by titration with zinc sulphate gives abnormal results owing to the precipitation of zinc as hydroxide, and this is also the case when the zinc sulphate solution is prepared as suggested by Procter ("Leather Industries Laboratory Book," pp. 55, 87), by adding ammonia to the solution until the precipitate just redissolves. If, however, an excess of ammonium chloride is added to the zinc sulphate solution (50 grms. per litre of $\frac{N}{10}$ solution), or if the ammonium chloride is added to the liquor to be titrated with Procter's solution, the precipitation of zinc hydroxide is prevented, whilst only zinc sulphide is precipated, and a sharp end-point of the reaction is indicated with lead acetate or sodium nitroprusside (preferably the latter) as an outside indicator. It is usual to take the alkalinity of a lime liquor as an index of its freshness, and to discard the liquor when the increase in alkalinity due to amino-compounds from the hides has reached a certain limit. In the presence of sodium sulphide, however, this direct measurement of alkalinity is not applicable; nor does the alkalinity of a sulphide lime liquor correspond to the separate alkalinities due to the sulphide and to the lime, a liquor containing sulphide showing less lime than pure lime liquor, owing to the sulphide precipitating calcium hydroxide.

C. A. M.

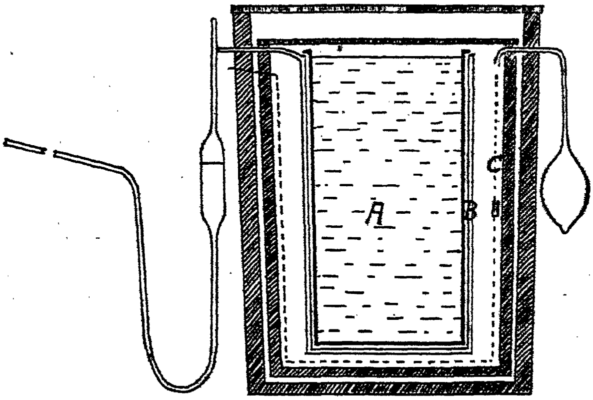
APPARATUS, ETC.



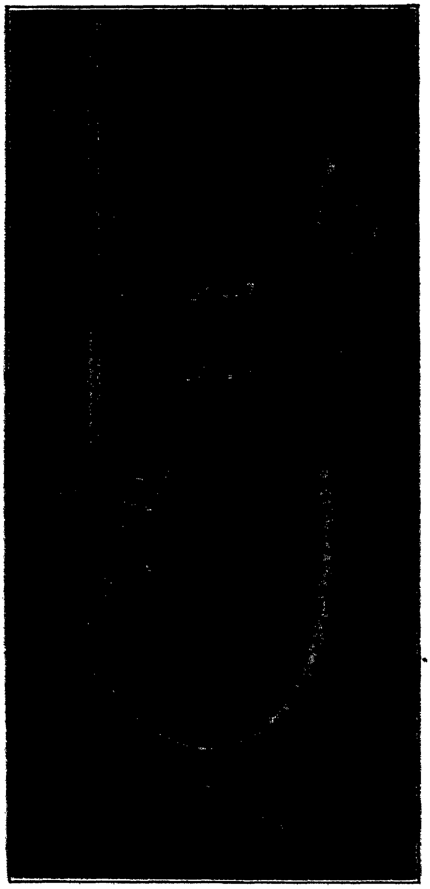
Modification of the Landsberger Boiling-Point Apparatus. J. H. Paterson. (*Proc. Phil. Soc. Univ. Durham*, 1912, **4**, 142-144.)—The author has simplified the apparatus of Landsberger (*Ber.*, 1898, **31**, 458). The boiling-flask *C* is partly filled with the pure solvent, and a few pieces of unglazed porcelain added to prevent bumping. The solvent is boiled with the tap *E* (of large bore) open, until the whole of the inner tube *A* is raised to the temperature of the boiling solvent, as shown by the condensation of the vapour from the condenser. The tap *E* is closed, when the vapour ascends by the side tube, and the measuring vessel *A* begins to fill with the liquid. When the thermometer registers a constant boiling-point, the burner below is removed and the tap *E* opened. The liquid collected above this tap flows back into the flask, causes a temporary cooling of its contents, and sucks the solvent in *A* back into the flask. A weighed quantity of the substance to be examined is next introduced into *A*, and the tap closed to such an extent that some vapour passes up through it and warms the outer jacket, while the rest passes by the side tube. When sufficient liquid has collected in *A*, and a definite temperature is noted, the tap *E* is opened, and simultaneously the tap *F* is opened to prevent the liquid being sucked back. The volume of liquid and the temperature are then noted, the tap again partly closed, and the experiment repeated at various dilutions. The apparatus gives good results. The thermometer has a range of 50° C., and is graduated in twentieths of a degree. The glass tap is greased with glycerol or vaseline according as benzene or alcohol is used as the solvent.

A. R. T.

Adiabatic Device for Bomb Calorimeter. J. A. Fries. (*J. Amer. Chem. Soc.*, 1912, **34**, 643-652.)—Adiabatic calorimeters have been described before (ANALYST, 1910, **35**, 283), but the present device is applicable to existing calorimeters of the more usual patterns. The calorimeter, *A*, is enclosed within and separated all round by a 4 to 5 mm. air space from a double-walled cylindrical copper jacket *B*, which has an air space of 4 mm. between its walls and bottoms, and is constructed of polished sheet copper of 0·25 mm. gauge. This copper jacket is in effect the bulb of an air-thermometer and is connected by a small brass tube, as shown, to a water-gauge which is shown—broken off—making an angle of 14° with the horizon, a capacious bulb being interposed between the gauge and the copper vessel to prevent water being sucked over into the latter should the temperature be unduly depressed. The fact that the air thermometer is sensitive to changes of barometric pressure is but rarely important, having regard to the short time required for a determination. The jacket is heated by means of an electric current passing through a German silver wire about 3·5 metres long and 0·8 mm. in diameter, with a total resistance of about 6 ohms. This wire is strung through brass eyelets screwed directly into the inner insulating fibre jacket of the Atwater-Berthelot calorimeter used by the author. The eyelets are so arranged that there is one coil of wire near the bottom a little distance from the sides, the rest of the wire going up and down along the sides, making eight loops spaced evenly, and coming to within 30 mm. of the top of the inner fibre jacket. The two ends are fastened to insulated copper wire and brought through the side of the fibre jackets. The manner of wiring described was adopted to avoid induction. The distance between heating wire and fibre vessel is about 6 mm., leaving 15 to 20 mm. between wire and calorimeter. On a 110 volt circuit, the current obtained through five 130 Watt lamps in parallel serves well. Since the air thermometer is affected by changes of pressure as well as changes of temperature its scale must be a movable one. This scale is graduated by placing a good mercury thermometer in the copper jacket, allowing the conditions to become constant, taking readings, then switching on the current for a short period and again taking readings when the conditions are constant, and so on, correcting for barometric changes if necessary. The scale of the air thermometer should be in 0·1°. With the author's instrument, adiabatic conditions are sufficiently realised if the heating device be put in operation when the charge is ignited and the current switched off after forty seconds, supposing, that is, that the charge is likely to generate some 4,500 calories. Whether another instrument would need a longer or shorter heating or one or two additional short contacts after the first can soon be determined. The author also finds that he gets the most exact results when his jacket temperature is maintained about 0·1 to 0·16° C. higher than that of the calorimeter, the explanation of this being probably the constant radiation from the water surface and top of the calorimeter. The advantages of the apparatus are these common to adiabatic



calorimeters generally—namely, obviation of tedious computation with some increase of accuracy. The following test numbers are given with some details of the experiments: Heat of combustion of benzoic acid, 6323, 6315, 6316, 6323, the best attested value known to the author being 6320. G. C. J.



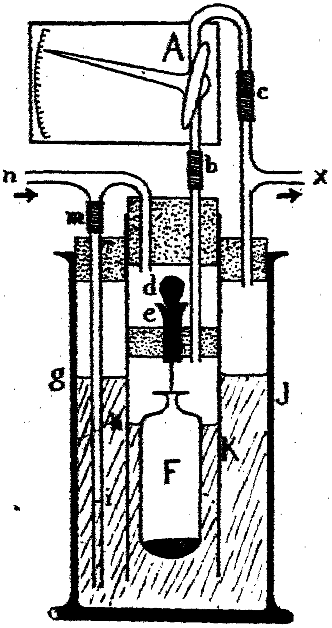
New Densivolumeter for Determination of Density of Solids. J. Escard. (*Comptes rend.*, 1912, 154, 1242-1244.)—The illustration is nearly self-explanatory. Before use the graduated tube (on the left) is raised and the other tube lowered, and the apparatus charged with water though *B*. By moving one of the tubes slightly up or down, the water level in *t* is made to coincide with the zero of the scale which is in $\frac{1}{20}$ c.c. A friction runner on *m* is then moved until it marks the level of the meniscus in the right-hand tube, which is then lowered until the bulb *A* is partly empty. The stopper *a* is removed, the weighed substance introduced, and the stopper reinserted. The levels of the tubes are readjusted until the meniscus in the right-hand tube again coincides with the position of the friction runner, and the volume of the solid introduced is read off direct on the graduated tube.

G. C. J.

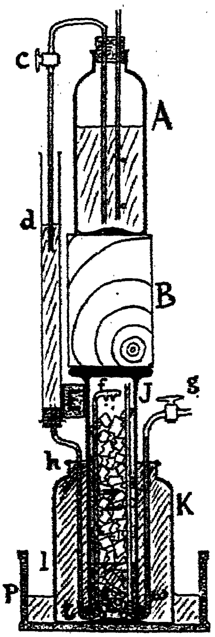
Apparatus for Determination of Vapour Pressure Isothermals of Silicic Acid Gels. R. Zsigmondy, W. Bachmann, and E. F. Stevenson. (*Zeitsch. anorg. Chem.*, 1912, 75, 189-197.)—The apparatus illustrated in the paper consists of (1) a small glass vessel for holding the gel, provided with a glass stopper, which is removed for the introduction of the gel, and which by a half-turn serves to make or break connection with the rest of the apparatus, from which it is detachable by a ground-glass joint; (2) a more capacious vessel for holding any liquid with the vapour pressure of which it is desired to bring the gel into equilibrium, this vessel being connected to a manometer, and also provided with a tap to disconnect its contents from the rest of the system; and (3) a T-tube connecting (1) and (2), the third limb of the T connecting through a tap to a mercury pump. For use with hydrogels, the taps and ground joints are greased for the upper part of their ground surface with "Ramsay grease"; but for use with alcohol or benzene grease is not permissible for the ground joint of the weighed vessel containing the gel, and mercury-cupping must be resorted to. As an example of the manner in which the apparatus is used, the following particulars relating to a series of experiments with a hydrogel of silicic acid are given. This gel was cut into pieces not larger than 4 mm. cube, and dried over concentrated sulphuric acid in an ordinary desiccator. The glass vessel for the reception of the gel was then weighed full of air, after removing every particle of grease from the stopper; the stopper was then greased, and the apparatus again weighed, exhausted, and weighed a third time. About 0·7 grm. of the gel was next transferred to the vessel, and the latter connected by its ground joint to the rest of the apparatus, the larger vessel, with manometer attachment, being charged with concentrated sulphuric acid. With the weighed vessel cut off by its tap from the rest of the apparatus, the rest of the apparatus was exhausted, connected momentarily with the weighed vessel, and the cycle repeated until, on opening the tap of the weighed vessel, the manometer remained stationary. The vessel containing the gel was then weighed daily, always closing its tap before disconnecting from the rest of the apparatus, and removing grease from its ground connection before weighing, until the difference between two successive weighings was less than 0·5 mgrm. Dry air was then admitted to the vessel and another weighing made. The concentrated acid in the vessel with manometer attachment was then replaced by acid of weaker strength, the system freed from air by the pump, and daily weighings again made, this series of operations being repeated with weaker and weaker acid, and finally with water. A reverse series, with increasing concentration of acid, was also made, and the results plotted on a system of rectangular co-ordinates, in which the ordinates are proportional to the vapour pressures, and the abscissæ to the water content of the gels in molecules of water per molecule of silica. The method, though tedious, is much less so than that of Van Bemmelen (*Zeitsch. anorg. Chem.*, 1897, 13, 239), and, unlike his method, can be applied to alcohol and benzene gels as well as to hydrogels. For use with benzene, sulphuric acid is replaced by mixtures of benzene with paraffin oil, whilst a mixture of glycerol and alcohol is used in experiments with alcohol gels. G. C. J.

Colorimeter for Rapid Work with Widely Varying Standards. C. H. White. (*J. Amer. Chem. Soc.*, 1912, 34, 659-662.)—The colorimeter is of the type in which the thickness of the sections observed is the variable. It consists essentially of two wedge-shaped hollow glass prisms of exactly equal dimensions, and open at the large end for the introduction of the solutions to be tested. The wedges are held in a vertical position side by side in a camera, and may be raised or lowered by rack and pinion, actuated by thumbscrews. The pinions are screened from view on the side towards the operator, except for a narrow horizontal slit across the middle of the camera, through which the solutions are observed when a test is being made. The carriers are graduated to correspond to the length of the wedges, the zero of the scale being opposite the index when the sharp edge of the wedge is opposite the narrow opening in the screen through which the colour is observed. The screens are adjustable, so that the opening may be varied to suit the operator. The ground glass shutter at the forward end of the camera, for diffusing the light, is hinged in the manner of a door to facilitate the transfer of the wedges to and from the camera. To carry out a determination with this instrument, equal quantities of the standard and of the material to be tested are diluted to equal volumes, and convenient amounts of the solutions are transferred to the wedges. The wedge containing the solution of unknown strength is set at the graduation representing the percentage—or some multiple of it—of the colouring matter in the standard. The wedge containing the standard is then adjusted, until the two fields seen through the camera appear identical. The percentage of colouring matter in the unknown is then indicated by the reading of the scale on the carrier containing the standard. If the depths of colour first compared are too great or too small for the most accurate comparison, the result may be checked, without changing the solutions, by setting the wedge containing the unknown at some new point on the scale, and again adjusting the other wedge until equality is reached. In the author's hands, the maximum error of a single observation is 0·6 per cent. The apparatus, which is illustrated in the paper, is made by Eimer and Amend, of New York. G. C. J.

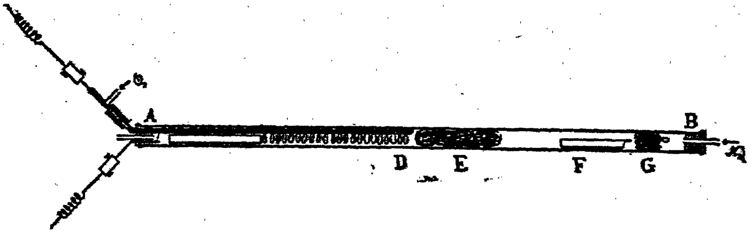
Combined Governor and Gauge for Regulating Flow of Gas. S. H. Collins. (*Proc. Phil. Soc. Univ. Durham*, 1912, 4, 110-113.)—The figure shows the governor and gauge for maintaining a flow of gas: *A* is a glass tap with pointer and scale; *b*, *c*, and *m* are rubber connections; *d* is a piece of glass rod drawn out to a long, fine point; *e* is a piece of glass Sprengel tube blown with a bulb and then ground down; *F* is a float containing mercury, and having a flat glass platform cemented on; *K* is a piece of wide glass tube, and *J* is a glass cylinder. The gas enters at *n* and leaves at *x*. When working the gas depresses the water to *i*, but instead of entering here, it passes by *d* into the chamber containing the valve. The rate at which the gas passes through this valve depends upon the position of *F*, which is kept hovering about a position determined by the level *h*, according to the size of the instrument. The level of water *g* is dependent on the amount of water placed in the jar, and may be adjusted at the commencement. The height *gh* is thus fixed; the height *gi* shows the pressures of the incoming gas, *hi* the surplus pressure being got rid of by the valve. As the difference of pressure on the two sides of the tap is kept constant, and the tap is fixed by experiment, both pressure and resistance are constant, and the current also in the absence of variation of specific gravity of the gas. The instrument in trial has shown an average flow of 10·05 ± 0·22 litres of gas per hour when 10 litres were required. A. R. T.



Constant-Pressure Hydrogen Generator. S. H. Collins. (*Proc. Phil. Soc. Univ. Durham*, 1912, 4, 107-109.)—The author claims that the apparatus delivers pure hydrogen at a steady rate. The figure shows a Winchester quart bottle, *A*, containing diluted sulphuric acid (1 : 8 by volume), and supplied with a stopcock, *C*. The wooden block *B* and the air-tube in *A* provide coarse and fine adjustment respectively for pressure of gas. The wide tube *d* is roughly graduated in c.c., the level of the sprinkler *f* being zero. The large bottle *K* serves to hold the waste acid, and in conjunction with the inverted cylindrical jar *J* acts as a gas-holder. The trough *P* receives the waste acid and is emptied occasionally. The jar *J* contains about 7 kgrms. of zinc in lumps (not granulated) resting on a ball of copper wire. Between the mouth of *J* and the bottom of *K* are two pieces of wide rubber tube to prevent breakage. The acid tube has a rubber connection at *t*, and the gas delivery tube similarly one at *w*, and these should be narrower than the tubes used as a cushion for *J*, to prevent their being closed by the weight of zinc. *hl* is only about three-quarters of *df*. Three corks are placed in the mouth of *K* to fix *J* firmly. The hydrogen is freed from sulphuretted hydrogen and other impurities by passing it through a tube containing "mercury lime," prepared by mixing 3 parts of mercuric chloride and 1 part of lime, making into a paste with water, drying the mixture, and breaking it into convenient sized lumps ready for use. This purifier gradually becomes yellow and then black by absorption of the impurities. A. R. T.



Analysis of Organic Substances by an Electrical Method. E. E. Reid. (*Amer. Chem. J.*, 1912, 47, 416-419.)—The following method is recommended for the simultaneous estimation of carbon, hydrogen, and sulphur. A combustion-tube of transparent quartz 600 mm. long by 17 or 18 mm. internal diameter is used, though one of hard glass may serve. In the end *A* is inserted a two-holed rubber-stopper, through one hole of which passes a small quartz tube, 300 mm. long by 4 mm. external diameter (*A* to *D*). At 25 mm. from the end this tube is bent to an angle of 45°, the bent end projecting from the stopper. To the outer end of this is fastened a T-tube, into the other side of which is fused a platinum wire. This wire must be



large enough to carry a current of 3 ampères without heating. The wire passes through the T-tube and small quartz tube, and connects at the end of the latter with one end of two metres of fine platinum wire ("No. 30") used as the heating coil; this is wound into a spiral about 12 mm. in diameter, and is supported on the small quartz tube. When hot, this has a resistance of 24 ohms, and is used on a 110-volt circuit, in series with an external resistance of 18 ohms, taking 2·6 ampères of current. This wire is coiled tightly round the small quartz tube for a distance of about 125 mm. from the stopper, and its end is joined to a rather stout platinum wire passing through the stopper. These close coils are so placed as to keep the lead peroxide in the boat just below them nearly, but not quite, up to its decomposition point.

A piece of platinum foil 100 by 25 mm. has welded across it, 1 mm. from the end, a piece of platinum wire. The other end is notched and turned up, and the foil bent to form a semicircular trough to fit inside the combustion tube. At *E*, just beyond the end of the small quartz tube, is placed a plug of quartz wool, 75 mm. long (asbestos will not answer, as it absorbs oxides of sulphur). Over this portion of the combustion tube is suspended a shield of thick asbestos board, and under it is placed a strong burner with a "wing" top. The plug of quartz wool at *G* contains a loop of platinum wire, to withdraw it when the boat is to be inserted. The portion of the tube containing the heating coil is provided with a close-fitting cover of asbestos board having a narrow slit through which the coil may be observed.

Lead peroxide is used as the oxidising material. A blank experiment, to determine its loss in weight when reduced to the monoxide, is made by heating a weighed quantity of the peroxide in the platinum boat in the combustion tube, using two burners under the tube and one from above. The peroxide is heated till it is at a red heat (3 mins.), when the source of heat is removed, and the boat cooled quickly. The resulting pure yellow monoxide is weighed, and the loss of weight calculated per grm. of peroxide. For an estimation, 1 to 2 grms. of the peroxide in the boat, spread out evenly, are placed in the combustion-tube, the quartz plug at *E* heated very hot, and the platinum coil brought to a bright red heat before inserting the boat containing the substance to be burned. Next the burner under the plug *G* is lighted, and a slow current of oxygen is admitted through the small quartz tube, care being taken that an excess of oxygen is present in the gases issuing at *A*. A third burner heats the boat at *F* to cause slow volatilisation of the substance, and the vapours are passed through the quartz plug at *E* by means of a slow current of air or nitrogen admitted at *B*. The most refractory substances are completely burned at the high temperatures possible in the quartz tube, admitting oxygen at *B* if necessary. 0·2 grms. of substance is burnt in about one hour, and even if complete combustion of the carbon is not assured, the sulphur is usually satisfactorily burned and absorbed. When the combustion is complete, the platinum boat containing the peroxide is heated to reduce it, as already described. Using the results of the blank, the loss in weight on reduction is calculated. The weight of the boat and its contents is diminished by this amount, and the remainder, subtracted from the final weight, gives the weight of sulphur trioxide absorbed during the combustion. A. R. T.

New Method for Quantitative Analysis of Solutions by Precise Thermometry. T. W. Richards and J. W. Shipley. (*J. Amer. Chem. Soc.*, 1912, 34, 599-603.)—The method depends upon noting the precise temperature at which the solution attains exactly the same density as a given, previously calibrated float, and its application is of course limited to cases where not more than one substance, in addition to the solvent, is present. When, as is usual, the solution is denser than the solvent, the more concentrated the solution, the higher is the temperature necessary to reach floating equilibrium; hence, each concentration corresponds to a definite temperature, and after a few points on the almost linear curve connecting the two variables have been determined by means of known solutions, all the intervening ones are determinable by simply reading the thermometer at the points where floating equilibrium is attained. When the solution is less dense than the solvent, it must be cooled instead of warmed to attain floating equilibrium, as the concentration increases. The exact density of none of the solutions need be known, since the method depends only on differences of density.

A mercury thermometer used for this purpose is best calibrated with great care; but if the bore is very uniform and the graduation is perfectly spaced, the degrees need not correspond to any recognised scale. Good results may be got with any good Beckmann thermometer. Platinum resistance thermometers may be used by those familiar with very exact electrical measurements, and in this case ohms or multi-volts may be plotted directly against concentrations without translation into Centigrade degrees. A thermostat capable of being adjusted to any needed temperature and of being kept there within 0·005° C. is required in order that the greatest accuracy may be attained. The solution to be investigated is placed in a litre Erlenmeyer flask, immersed in the thermostat and the float within the flask is viewed by reflection in a small inclined mirror kept beneath the water of the bath. The glass float should be small (5 c.c.) and cigar-shaped and, if permanency in calibration is desired, should have been made several months before use. A recently-made float may be used, but will require calibration every few days. For wide ranges of concentration, several floats will be necessary. Because the co-efficient of expansion of the float enters into the effect, the results obtained with floats made of different materials are not directly comparable. Probably Jena thermometer glass (59^{III}) is the best material available. The exact density of the float is of no consequence: all that is necessary is that it shall float at room temperature in the lightest solution for which it is to be used, and sink when the temperature is raised a few degrees. A float of slightly too low density is easily adjusted by attaching to it a few points of glass. The sensitiveness of such floats is very great and the temperature at which they sink or rise can be determined within 0·001° C. As an example of the order of magnitude of the temperature differences corresponding to differences of concentration which the method is designed to measure, the following may be given: A particular float just sank in alcohol of 98·99 per cent. strength at 15·391° C. On adding water so that the percentage of water in the alcohol was increased from 1·01 per cent. to 3·44 per cent., the temperature of floating equilibrium was 24·093, and intermediate experiments showed that the increase of temperature of floating equilibrium was very nearly a linear function of the concentration of water. It is obvious that this float affords a means of determining the percentage strength of strong alcohol with a degree of accuracy which is limited rather by errors inherent in the methods available for determining the strength of the 98·99 per cent. spirit used as the basis of calibration than by the experimental errors of the method now described.

G. C. J.

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REPORT.

The Softening, Purification, and Sterilisation of Water Supplies. A. C. Houston. (Metropolitan Water Board. Eighth Report on Research Work.) —The report deals with a novel method of using lime primarily as a bactericidal agent, and only secondarily and incidentally for softening purposes. This would make possible the safe and speedy utilisation of flood-water, and the absolute elimination, so far as the portion of water treated with an excess of lime is concerned, of any danger from such epidemic water-borne diseases as typhoid fever and cholera. As an illustration of the method, it is assumed that it is required to deal with 10,000 gallons of the Thames supply, to remove at least 15 parts of total hardness, and to render the water innocuous. Fifteen pounds of quicklime, costing 1½d., would be added to 7,500 gallons of raw, unstored Thames water. This would kill within twenty-four hours the *B. coli*, and inferentially, but certainly, the microbes also of epidemic water-borne disease—*e.g.*, the typhoid bacillus. The water would also be improved considerably as judged by physical and chemical standards. The excess of lime (about 0·007 per cent.) would then have to be neutralised with 2,500 gallons of *adequately* stored water, which, according to all the author's experiments, would not contain any of the microbes of epidemic water-borne disease. Thus 75 per cent. of the water would be sterilised chemically, and the remaining 25 per cent. by storage. The mixture would have lost about 15 parts of its total hardness, and would contain no undesirable excess of free lime, besides being perfectly innocuous. Rapid filtration alone would then be required to remove the precipitate of inert carbonate of lime, and to bring the water up to a reasonable standard of chemical purity. As an alternative, the case of a well water is considered, having over 15 parts of temporary hardness, which is considered to be dangerous. Seventy-five per cent. of the whole would be lime-sterilised, and the remaining 25 per cent. sterilised by ozone or heat, or by active chlorine, and the two portions mixed together to produce a softened and bacteriologically safe drinking-water. As only 25 per cent. of the total would require the more expensive treatment, the cost is proportionately reduced, and the faint taste in the 25 per cent. portion would be diluted to such an extent as to be negligible. Experiments are cited showing that the bactericidal dose of lime with hard waters is about 1 to 5,000, falling in the case of very soft waters to about 1 to 50,000. The experiments were made with "uncultivated" *B. coli*—the state in which they would be ingested by the consumer. The *B. coli* in London crude sewage were killed by 1 part of quicklime (about 75 per cent. CaO) in 2,000 parts of sewage. The disadvantages and the advantages (of the excess lime method) are set out, and among the former, assuming the method to be grafted on to an existing water-purification plant, is the fact that the mere cost of lime to reduce the hardness, even by 10 parts, would be about double the cost of the existing process of sand-filtration. The capital expenditure on the machinery and softening tanks would be very large, and the periodical removal and disposal of sludge would entail great expense, and might even be impracticable. Softening proposals (apart from the novel application of utilising the lime as a germicidal agent) would seem to run counter to the authoritative "findings" of the "Richmond," "Balfour," and "Llandaff" Commissions, and of the Arbitration Court. As against all these, the method can be applied to 75 per cent. or any smaller proportion of the total supply, and is specially suitable for flood-waters, because the water is then usually less hard, and the treatment has a clarifying and purifying action, apart from its bactericidal effect. The cost of lime treatment is not wholly unremunerative, and it is even claimed by some that a substantial gain on balance is obtained, due to saving in soap and fuel consumption. Of first-rate importance is the capacity of the method quickly (five to twenty-four hours) to render an initially foul water absolutely safe bacteriologically; it also virtually increases the yield of the sources of supply by rendering them always fit for abstraction. The method is perfectly innocuous, since no substance is left in the water which was not there initially in greater amount. The construction of new storage reservoirs could be postponed, as the lime-treated water is brought into a parallel bacteriological condition with the *adequately* stored water. This would mean, in effect (on the 75 per cent. basis), prolonging the period of storage from 30 to about 120 days, since 25 per cent. of the water from the storage reservoirs could be mixed with 75 per cent. of raw water after lime-sterilisation.

H. F. E. H.

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REVIEWS.

METHODS OF AIR ANALYSIS. By J. S. HALDANE, M.D., F.R.S. Pp. x + 130. London: Charles Griffin and Co. 1912. Price 5s. net.

Most of those who are actively engaged in air analysis are more or less familiar with the work of Dr. Haldane in this direction, and to these it is enough to say that this little book contains illustrations and detailed instructions for the use of all those pieces of apparatus which we owe to the author—illustrations and instructions which until now were hard to come by, as they were scattered among a number of journals not usually to be found on the analyst's shelves and among Bluebooks. To those whose general knowledge of the subject was acquired a few years ago, and who have not given close attention to more recent periodical literature, it may be said, without exaggeration, that the work of Haldane has revolutionised methods of air analysis—at any rate of methods designed to estimate three or a less number of constituents in a gas mixture. Haldane's original apparatus was described as long ago as 1898, but since that date various modifications of it have been devised for special purposes, and perhaps the best known is his apparatus for the gasometric estimation of carbon dioxide in the air of ordinary rooms, and this apparatus serves as well as any to illustrate the principles common to all.

For the gasometric estimation of the small proportion of carbon dioxide usually found in ordinary rooms and factories, a portion at least of the measuring burette must be of very narrow diameter, and a feature of all apparatus of the Haldane type is a burette with a bulb of relatively large dimensions, and a narrow, graduated stem. The relation between the volume of the graduated stem and the bulb differs according to the purposes the apparatus is intended to fulfil; but for the determination of carbon dioxide in air the stem makes up only one-hundredth of the volume of the whole burette, and is divided into 100 divisions. Since the scale is sufficiently open to allow fractions of a scale division to be estimated with an error not exceeding 0·1, very close reading is possible; but it is obvious that such close reading would be futile unless there existed means for correcting for variations—even very small variations—of temperature and pressure between two readings. An essential feature of the Haldane apparatus is that it provides such a means of compensating for variations of pressure or temperature. The means adopted is a control-tube, of the same size and shape as the burette, placed alongside the latter in a water jacket, the burette and control-tube being connected by independent connections to separate limbs of what is in effect a differential pressure gauge, charged with a solution of caustic potash. Dr. Haldane, in his book, acknowledges the use of a control-tube by Williamson and Russell, and the use of the same principle by Pettersson; but the application of the principle in a simple and practical form is due to Dr. Haldane alone, as is constantly recognised by designers of new forms of gas apparatus when, to shorten their descriptions, they say their apparatus embodies the Haldane principle.

Since the appearance of the publications in which Dr. Haldane's various pieces of apparatus were first described, many minor improvements have been introduced, and some of these were described in a paper by Butterfield (ANALYST, 1909, **34**, 257)—a paper which had a special value at the time because of the inaccessibility to many of Haldane's original papers. All these minor improvements are referred to in the present book, which will therefore be welcomed even by those familiar with the original papers.

In addition to gasometric methods, the book includes a description of the Haldane and Pembrey method for the gravimetric determination of moisture and carbon dioxide—a method which, by simple means, enormously increased the speed and accuracy of such determinations—and modern methods for the recognition and determination of small proportions of carbon monoxide in air are also dealt with at length. In this connection, again, the work of Haldane (and Douglas) has at once simplified and increased the accuracy of the blood test, the use of the spectroscope being dispensed with and the delicacy of the test increased about fivefold.

This book, which, in addition to its other merits, is well illustrated and exceptionally free from misprints, must find a place in the library of every chemist who is at all interested in the subject with which it deals, and those chemists who are not interested in the analysis of simple gas mixtures must be few.

G. CECIL JONES.

An Introduction to Quantitative Analysis. By S. J. M. Auld, D.Sc. (Lond.), Ph.D., F.I.C. London: Methuen and Co., Ltd. 1912. Pp. x + 215. Price 5s. net.

This is one of Methuen's Textbooks of Science series, and aims at providing "an introductory course in quantitative analysis, suitable for college and university students, without entering too deeply into the details of special technical analysis," and in this object it succeeds. That it does so to an appreciably greater extent than many of the existing similar handbooks is open to doubt. Little can be said in favour of the practice of multiplying elementary textbooks, which do not efficiently replace good laboratory teaching, which do not particularly encourage independent ingenuity on the part of the student, or which fail to emphasise the scientific foundations of analytical chemistry.

The author has traversed practically the whole range of ordinary analysis, and has been in this respect too ambitious; for instance, the accounts of water and gas analysis are inevitably scrappy. No doubt the actual manipulative detail of water analysis may be properly described in seventeen pages, and it may be argued that the really important thing, which is the interpretation of the results, can be taught by experience alone. This branch of analysis is surely better left to the special technical treatise.

As to the methods and exercises properly included in a college course, the selection in the present volume is satisfactory. It is to be regretted that the use of standard thiocyanate is omitted, and that no reference is made to the volumetric estimation of mixed alkali, hydroxide, and carbonate, but, on the other hand, one is pleased to see borax brought into line with other weak alkalis.

The book is reasonably free of errors and misprints, but grave exception will be taken to the explanation of bichromate oxidation by the scheme—

$$Cr_2O_6 \longrightarrow Cr_2O_3 + 3O, \text{ on p. 58.}$$

In the estimation of sulphur by means of sodium peroxide, the possible injury to a platinum vessel should be indicated, and in the determination of the equivalent of magnesium by heating in air, mention ought to have been made of the formation of magnesium nitride, a point that often puzzles a thoughtful student. The weighing of a precipitate on a "tarred" filter-paper (p. 125) points to a somewhat hasty revision. Apart from these criticisms, which after all might be directed against most elementary books, this volume has the merit of brevity, and will no doubt be of use to the students for whom it is intended. G. Druce Lander.

Sulphuric Acid and Alkali. By G. Lunge. Vol. iii.: Ammonia Soda, Various Processes of Alkali Making and the Chlorine Industry. London: Gurney and Jackson. 1911. Price 30s. net.

The analyst who may have to refer to the third volume of Dr. Lunge's great work on alkali industries in search of detailed information respecting special analytical methods will be disappointed. For these another volume by the same author must be consulted; but the present volume contains a number of tables of specific gravities and strength of solutions, as well as sufficient analytical information for carrying out the various operations in a factory. Many tables of statistics are given which might have been useful had they been brought up to date. The production of ammonia soda, for instance, is only given to 1895, and can only possess historical interest sixteen years later. There are numerous indications throughout the book that it has neither been carefully revised nor brought up to date since the issue of the second edition in 1896. As the author remarks, even the description of many antiquated or impracticable processes may be of occasional use to the professional reader; but still those who refer to a work by a technical expert expect up-to-date information as to the actual working of factories and a little more guidance than a mere enumeration of processes because they have been patented. The reader who is not already an expert will have difficulty in ascertaining what the best practice is at the present time in factories producing the materials referred to in the work. For instance, Schlœsing's ammonia soda process of 1889 is called new, and the same term is applied to a patent taken out in 1893 and long since expired.

The bulk of the volume might, with advantage to the reader, be reduced by at least one-third, and it is to be hoped that in any future edition the editing will be more carefully done and the matter brought up-to-date. Errors are very numerous, and in some cases misleading. We are told, for instance, that the American short ton is 1,000 pounds, and in a description of apparatus on p. 121, a drum is substituted for a chain. Even the reprint of a German title contains two errors in one line.

The 184 illustrations are clear and well executed, but many of them are mainly of antiquarian interest to the chemical technologist of to-day. WALTER F. REID.

NITROCELLULOSE INDUSTRY. By E. C. WORDEN. 2 vols. London: Constable and Co., Ltd. 1911. London. Price 42s. net.

The compilation of this book has been a work of very great magnitude. It is impossible, in the course of a short review, to do anything approaching justice to the results of the author's industry. The text runs to over 1,000 pages, the references total nearly 18,000, some 500 different journals have been consulted, and the patent specification references are international, and are inclusive of Japan and Mexico. The reviewer has been particularly struck with the reliable and up-to-date nature of the information in those sections of the book which deal with gun-cotton and propellants. The analyst will find many subjects very fully dealt with. Fusel oil and amyl acetate are treated at considerable length from the manufacturing point of view, and the analysis of amyl acetate, crude fusel oil, and purified amyl alcohol are dealt with very adequately. The section on camphor, artificial camphor and camphor substitutes, is a very important one. Very full details are given of the methods adopted for the analysis of lacquers, celluloid, photographic films, and pharmaceutical collodion. The article on artificial filaments is especially worthy of note, as it deals with the subject in a most interesting manner. Thirty pages are devoted to the United States regulations for denatured alcohol. The book, as a whole, claims the highest commendations, and it can certainly be consulted with very great profit by everyone who has to deal with nitrocellulose, either in its wide ramifications in the arts, or in its use in explosives. GEORGE W. MACDONALD.

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ON THE APPLICATION OF ADSORPTION TO THE DETECTION AND SEPARATION OF CERTAIN DYES.

BY A. CHASTON CHAPMAN AND ALFRED SIEBOLD.

(*Read at the Meeting, June* 5, 1912.)

DURING recent years a very considerable amount of attention has been devoted to the study of certain phenomena to which the general term "adsorption" has been applied. Whatever may be the precise explanation of these phenomena, the facts are comparatively simple, and many instances—*e.g.*, the decolorisation of solutions by means of animal charcoal—have been known and utilised for a very long time. Among adsorbent substances, kaolin occupies an important position, and a number of references to its power of removing dyestuffs from solution are to be found in chemical literature. These, as a rule, occur in communications dealing with the theory or practice of dyeing, and, with the exception of the valuable work of Goppelsroeder on the separation and identification of certain colouring matters by capillary methods, no systematic study of this property, from the analytical standpoint, has, so far as we are aware, been made. It is true that H. Wislicenus (*Zeitsch. Chem. Ind. Kolloide.*, 1908, 2, Supt., II., 11-20) has suggested the application of the so-called "fibre-alumina" to the examination of various kinds of extracts, including dyes, and that LaWall has employed kaolin as one of the reagents in a scheme for the detection of certain natural colouring matters ("Select Methods of Food Analysis," Leffmann and Beam, 2nd Edition, pp. 74-75), but no very definite results appear to have been obtained, and no mention is made of the separation and identification of artificial dyes. Among the more important papers dealing with the question of adsorption of coal-tar dyestuffs, attention may be specially directed to a paper by Suida (*Monatsh. Chem.*, 1904, 25, 1107), in which the adsorbing properties of kaolin and other silicates and silicious materials have been studied.

Few problems are more difficult than the identification of the dyes used in the colouring of foodstuffs, particularly when two or more are present together, and no apology is needed for bringing to the notice of analysts any method which is capable of facilitating such investigation, even if only to a small extent. The experiments described below had their origin in some observations made in the laboratory of one of us, that certain artificial dyestuffs could be readily separated by the use of kaolin, and that this property could in some cases be usefully applied to the examination of artificially coloured food-products.

The method of experimenting was very simple, and consisted in stirring in a small glass mortar the solution of the dye with a weighed quantity of kaolin, previously ground with a little water. Alternatively the dry kaolin was added to the dye solution, and the mixture thoroughly stirred, or in some cases the solution of the dye was merely filtered through a layer of kaolin. The dye solutions contained in all cases 1 grm. of the dyestuff per litre. For the purposes of the experiments, 10 c.c. of the dye solution were stirred for several minutes with 5 grms. of kaolin, previously mixed with 10 c.c. of water, and the mixture filtered through a Buchner funnel with the aid of a pump. The above amount of kaolin was always employed, but in the case of those colours which are very rapidly adsorbed, it was found that the kaolin was capable of taking up in some cases more than 1 per cent. of its weight of the dye. When tested in this manner, it was found that the various dyes experimented with could be divided into three classes: (*a*) those which are entirely adsorbed under the above-mentioned conditions; (*b*) those which are partially adsorbed, giving coloured kaolin from which the bulk of the dye can be extracted by washing with water; and (*c*) those which are not appreciably adsorbed.

The following table contains a list of the dyes experimented with, and shows their behaviour when treated in the manner above described:

TABLE I.

Adsorbed.	Partially Adsorbed, but Adsorbed Colour partly extracted by Washing with Water.	Not Adsorbed.
Congo red.	Acid green.	Acid magenta.
Safranine.	Patent blue.	Eosin.
Magenta.	Soluble blue.	Erythrosin.
Neutral red.	—	Fluorescein.
Malachite green.	—	Methyl orange.
Brilliant green.	Acid green. (Practically all extracted by boiling water.)	Tropæolin.
Methylene blue.	—	Orange IV.
Bismarck brown.	—	Ponceau 4R.
	—	Bordeaux R.
Crystal violet.	—	Sudan red.
Methyl violet B extra.	—	Tartrazin.
Auramine.	—	Naphthol yellow.
		Picric acid.
		Naphthol green.
		Indigo carmine.
		Cochineal (1 per cent. solution).

Although three entirely different lots of kaolin which were experimented with behaved very similarly so far as their powers of adsorption were concerned, yet slight differences were observed, particularly in the case of certain of the partially adsorbable dyes.

A convenient method of ascertaining whether any particular sample of kaolin is suitable for the examination of dyes by this method, is to mix 10 c.c. of a 0·1 per cent. aqueous solution of naphthol yellow, and 5 c.c. of a 0·1 per cent. aqueous solution of soluble blue. In the case of a really good kaolin, 5 grms. should be sufficient for the complete separation of these two colours—that is to say, the filtrate should be yellow and not tinged with blue. Satisfactory separations of the following dyes were effected :

Methyl violet B extra and naphthol yellow, crystal violet and naphthol yellow, crystal violet and eosin, auramine and naphthol green, auramine and indigo carmine, auramine and acid magenta, magenta and tropæolin, methylene blue and cochineal (1 per cent.), malachite green and tropæolin, brilliant green and tropæolin, methylene blue and tartrazin, crystal violet and methyl orange, soluble blue (5 c.c. only) and naphthol yellow.

In some cases it happens that, although a dye is itself readily soluble in alcohol, it is yet incapable of being dissolved from the coloured kaolin by means of that solvent. It is clear, therefore, that this property might in some instances be made use of in order to effect the approximate separation of three dyes when present together. Thus, the 5 grms. of kaolin, having been shaken or stirred with the dye solution in the manner indicated above, and filtered, the coloured kaolin remaining on the filter might, after one or two washings with hot water, be treated with hot alcohol. The following are examples of separations effected in this manner :

TABLE II.

Ponceau 4 R:	Aqueous filtrate.	Red.
Crystal violet:	Alcoholic filtrate.	Purple.
Safranine:	Kaolin.	Pink.
Naphthol yellow:	Aqueous filtrate.	Yellow.
Magenta:	Alcoholic filtrate.	Red.
Methylene blue:	Kaolin.	Purplish-blue.
Fluorescein:	Aqueous filtrate.	Dirty pink and fluorescent.
Brilliant green:	Alcoholic filtrate.	Green.
Congo red:.	Kaolin.	Pink.

That the coloration of the kaolin in the above cases was, in fact, due to the safranine, methylene blue, and congo red respectively, was proved by the application of well-known tests.

Experiments were next made for the purpose of ascertaining to what extent the adsorption results given above apply to saccharine solutions. In the case of magenta, methyl violet, safranine, Bismarck brown, brilliant green, auramine, and soluble blue, the results were substantially the same as when sugar was absent. The above results apply entirely to the removal of dyes from *aqueous solutions* by means of the kaolin, but in many cases the removal of the dye from alcoholic solutions can be

TABLE III.

Kaolin Dyed by Adsorption of—	H_2SO_4.	H_2SO_4, 10 per cent. by weight.	HCl.	HNO_3.	NaOH, 10 per cent.	NH_4OH.	Alcohol.
Congo red.	Deep blue.	Slate blue.	Deep blue.	Bluish-slate.	No change.	No change.	Alcohol, tinted light pink.
Safranine.	Green, changing to dull violet on dilution.	Purple.	Greenish-blue.	Bluish-green.	„	„	Alcohol, coloured light pink.
Magenta.	Brownish-yellow.	Brownish.	Yellow.	Yellowish-brown.	„	Slowly becomes lighter.	Deep pink solution.
Neutral red.	Green.	Violet-blue.	Blue.	Bluish-slate.	Yellow-brown.	Yellow-brown.	Little change; reddish-yellow filtrate.
Methyl violet B extra.	Yellowish-brown.	Fawn.	Brownish-yellow.	Greenish.	Lighter and duller.	No change.	Violet solution.
Crystal violet.	Brownish-yellow.	Greenish.	Orange-yellow.	Dull orange.	No change.	„	„
Malachite green.	Yellow.	Fawn.	Bright yellow.	Yellow-red.	Nearly decolorised.	Decolorised.	Greenish solution; partly extracted.
Brilliant green.	Orange.	Yellowish-brown.	Yellow.	Yellowish-red.	„	„	Deep green solution; easily extracted.
Methylene blue.	Yellowish-green, becoming blue on dilution.	No change.	No change.	Bluish-green, becoming blue on dilution.	No change.	No change.	No action.
Bismarck brown, or Vesuvin.	Puce.	Darker.	Puce.	Puce.	Browner.	Yellower.	Yellow on boiling.
Auramine.	Fawn.	Much lighter.	Decolorised.	Decolorised.	Decolorised.	Lighter.	Yellow solution.

effected by the same method, although not in the same degree. Thus, working under the above conditions, safranine which is completely removed from water, was only partly adsorbed from a solution in 90 per cent. alcohol. The kaolin containing the adsorbed dyes can be tested by the application of reagents similar to those which have been employed for the detection of dyes on wool and other fibres. As we have verified these reactions in the case of the colours experimented with, it may be useful to give the table (p. 342), although the reactions are in many cases to be found in various textbooks dealing with the subject:

The coloured kaolins were, in all cases, dried before the application of the above-mentioned reagents. The dilute sulphuric acid referred to was prepared by mixing one part by weight of the acid with nine parts of water; the sulphuric acid, hydrochloric acid, and nitric acid were the ordinary pure laboratory reagents. The caustic soda solution was a 10 per cent. solution, and the ammonia was prepared by mixing one volume of the strong ammonia solution with two volumes of water. The alcohol referred to in the last column was 90 per cent. strength.

It will be seen that we have only dealt with a few of the more commonly occurring dyestuffs, but doubtless the method would prove applicable in the case of a great many other colouring matters with which we have not experimented. It may be said that no difference could be observed in the adsorbing properties of kaolin when this was extracted with dilute hydrochloric acid and thoroughly washed. On the other hand, the ignition of the kaolin seemed to weaken its adsorbing properties to some extent. Precipitated aluminium hydroxide ground up with water (the so-called alumina cream) was found to be very inferior to kaolin as an adsorbent.

DISCUSSION.

Mr. BEVAN asked whether the respective proportions in which the dyes were present would make any difference in the accuracy of the test, and also whether there appeared to be any connection between the chemical composition of the various dyes and their degree of adsorption.

Mr. RAYMOND ROSS asked whether kaolin had any action upon natural vegetable colouring matters, such as occurred in fruits and in jam.

Mr. J. CONNAH asked whether this method had been applied to the identification of vegetable colours (*e.g.*, fruit juices) as well as to the coal-tar colours, and remarked that the method would doubtless be useful for the detection of artificial colouring matters in wines.

Mr. E. R. BOLTON asked whether the method could be used to separate colours from oils, and also whether the authors had tried the use of Fuller's earth. He suggested that possibly the use of kaolin and Fuller's earth consecutively might be found advantageous. Fuller's earth, of course, varied a good deal in its action on oils, some samples being capable of bleaching oils that would not be affected by other samples.

Mr. H. E. BURGESS said that he had recently found that when a solution containing three or four of these dyes was applied to ordinary blotting-paper the colours were separated, and could be fairly accurately identified by cutting up the blotting-paper and dissolving out the separate colours. The colours in that case were dissolved in glycerol before being applied to the blotting-paper; when water was used, the same effect was not obtained.

Mr. **Evershed** said that those colours which were adsorbed by kaolin were all, or nearly all, basic colours, whereas those which were not adsorbed were "acid" dyes. The action of the kaolin was therefore analogous to that of animal fibres. A skein of wool would take up the basic colours from a neutral bath, while the acid colours would only dye wool in the presence of a little mineral acid. He should like to ask whether the authors had tried the effect of mineral acids upon the adsorption of any of these dyes by kaolin. Had any quantitative tests been made with mixtures of acid and basic colours to see whether the former passed through the kaolin completely? It was well known that if fairly strong solutions of acid and basic colours were mixed, a precipitate was generally formed. If this precipitation occurred with weak solutions in passing through the kaolin, the separation must be incomplete. Had the kaolin method any superiority over the well-known methods of testing by means of wool and cotton?

Mr. L. M. **Nash** asked whether this method applied to spirit-soluble colours.

Mr. **Richmond** said that a good many years ago a method was published for detecting artificial colours in butter and margarine by shaking up with "Spanish earth," which, while not affecting the natural colouring matters, removed a good many artificial colours.

The **President** remarked that the fact that it was possible, when two out of a mixture of three dyes had been adsorbed by kaolin, to remove one of these by treatment with alcohol, showed how great an influence was exercised by the liquid in which the colours were dissolved. No doubt, if alcohol had been used in the first instance, different effects as regards adsorption would have been produced, and it might perhaps be possible, by varying the solvent, to extend the application of the method.

Mr. **Chapman**, in reply, said that the classification of the various dyes into those which were adsorbable by kaolin and those which were non-adsorbable was not, of course, an absolutely sharp one, but the method was at least capable of permitting of the separation of a number of dyes from one another, and of rendering their identification possible. It was true that, generally speaking, the dyes which were adsorbable by the kaolin were those which had a basic character, whilst those of an acid character remained, as a rule, unadsorbed. This had been pointed out some years ago by Suida and others. It was, of course, also true that some of the dye stuffs, to which he had referred, interacted and produced some precipitation. One advantage of the kaolin method over the ordinary one of dyeing wool, and also over such capillary methods as those mentioned by Mr. Burgess, was that larger quantities of the dyes could be separated, and that the dyed kaolin could be tested by means of various reagents, which could not well be applied to wool or paper. He and Mr. Siebold had not devoted much attention to natural colouring matters, but a good deal of work had already been done on that part of the subject, and had been referred to at the beginning of the paper. They had had no experience of the application of this method to the testing of wine, but it seemed quite possible that it would enable certain artificial dyes to be separated from the natural colouring matters of the wine. In the case of alcoholic solutions, separation could in many cases be quite readily effected, although they were of a different character from those which had been described in the case of aqueous solutions. They had not made any experiments with oils, and could not say what results would be obtained.

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A NEW METHOD FOR THE DETECTION AND ESTIMATION OF SMALL QUANTITIES OF NITROUS ACID.

By E. HOLL MILLER.

(*Read at the Meeting, June* 5, 1912.)

THIS method is a colorimetric one, and depends on the formation of paranitrosodimethylaniline, when a solution of dimethylaniline hydrochloride is added to an acidulated solution containing nitrous acid. A yellow colour is developed immediately, unless the nitrous acid solution is very weak, in which case the colour develops on standing.

The following process is recommended for carrying out the estimation: A solution of dimethylaniline hydrochloride is required, containing 8 grms. of dimethylaniline and 4 grms. of hydrochloric acid per 100 c.c., and a standard solution of sodium nitrite containing 1 part of nitrous acid per 100,000. The estimation is performed in Nessler cylinders, or a colorimeter. Fifty c.c. of the solution under examination are introduced into a cylinder, and acidulated with 1 drop of concentrated hydrochloric acid, and 3 drops of the dimethylaniline solution added, and allowed to stand fifteen minutes. If the solution is very weak, it may be necessary to allow it to stand for thirty minutes, or longer, for the complete development of the colour. The colour is then matched in the usual manner against the standard solution, which is acidulated with 1 drop of concentrated hydrochloric acid, and 3 drops of the dimethylaniline solution added. The method is very sensitive, and will detect with ease 1 part of nitrous acid per 1,000,000. The presence of nitrates does not interfere with the estimation. The great advantages of the method are its sensitiveness and simplicity.

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NOTE ON THE DETECTION OF BENZOIC ACID IN MILK.

By CECIL REVIS.

THE use of benzoates for the preservation of milk has shown a considerable and wide-spread increase this year, though they seem to be seldom detected. This is undoubtedly due to the fact that benzoic acid is not usually looked for in a routine manner, and also, that the quantity of milk taken for official purposes is, as a rule, too small to allow of the examination when other tests have been made. As a further difficulty arises from the unreliability of some of the methods employed, it may be of interest to note that the following method gives certain results. While not entirely original, it embodies the most necessary procedure in the detection of benzoic acid, viz., that the removal of fat, protein, etc., shall be done in an alkaline medium, otherwise there is much liability of the benzoic acid being removed with the precipitate or coagulum. The method is as follows:

One hundred c.c. (not less) of milk are diluted with an equal volume of water, and, after the addition of 5 c.c. of 10 per cent. sodium carbonate solution, heated in boiling-water for two to three minutes; 10 c.c. of 20 per cent. calcium chloride solution are then added, and the heating continued, until coagulation of the casein, etc., is complete. The liquid is then cooled and filtered, and the filtrate, neutralised with hydrochloric acid to litmus-paper. Ten c.c. of Fehling copper sulphate solution (not mixed with the tartrate solution), followed by 10 c.c. of a solution of potassium hydrate (containing 31·18 grms. per litre) are now added, and the liquid again filtered. The filtrate is poured into a separating-funnel, acidified with hydrochloric acid, and extracted with about 50 c.c. of ether. The ether is then washed three times with a little distilled water. About 10 c.c. of water are now added to the ether in the funnel, together with 1 drop of phenolphthaleïn solution, and then a saturated solution of barium hydrate added gradually, until, on violent shaking, the aqueous layer remains pink. This is then filtered off into a porcelain basin and evaporated to about 5 c.c. The contents of the basin are filtered into a test-tube and dilute (1 in 100) acetic acid dropped in until the pink colour is discharged, after which two more drops are added. The liquid is then tested with 1 drop of 10 per cent. neutral, freshly-prepared solution of ferric chloride, when, in the presence of benzoic acid, the usual characteristic precipitate is obtained. This method will detect 0·02 per cent. of benzoic acid.

The test with ferric chloride is the most reliable and characteristic, but it is necessary that everything else shall, as far as possible, have been removed, which the above method ensures.

When examining cream, 50 c.c. should be diluted to 200 c.c. with water and the same treatment applied.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

ANALYSIS OF FOOD AND DRUGS.

Examination of Copaiba Oil. E. Deussen and B. Eger. (*Chem. Zeit.*, 1912, **36**, 561-562.)—On treating β-caryophyllene with nitrogen peroxide, a compound, $C_{12}H_{19}N_3O_6$, which may be termed β-nitro-caryophyllene (m.-pt. 159·5° to 160° C.), is obtained. This reaction may be used in the examination of copaiba oil in the following manner: Three grms. of the sample are dissolved in 27 c.c. of anhydrous ether, and the solution treated with nitrogen peroxide until the precipitated β-nitro-caryophyllene begins to agglomerate. The precipitate is then separated, washed with ether, dried on porous porcelain, and weighed (*cf. Annalen*, 1912, **388**, 138). The following yields of β-nitro-caryophyllene were thus obtained: Caryophyllene (from clove oils) 50 to 52; Para copaiba oil (*a*), 9·5 to 10; (*b*, *c*, *d*,) 15 to 16; Maracaibo copaiba oils, 3 to 6; Maranham copaiba oil, 8 to 9; Para copaiba oil (*d*) adulterated with 10 per cent. of gurjun oil, 13·3 to 14·3; with 20 per cent. of gurjun oil, 11·7 to 12·7; with 30 per cent., 10·7 to 11·7; and with 50 per cent., 7·7 to 8·3 per cent. For the detection of 10 per cent. of African copaiba oil in Para oil, the optical rotation may be determined. The melting-point of the dihydrochloride also rises with the increase in the proportion of African copaiba oil. Thus, on passing hydrochloric acid gas into pure Para oil, a mixture of β-caryophyllene dihydrochloride (m.-pt. 69° to 70° C.) and of *l*-cadinene hydrochloride (m.-pt. 117° to 118° C.) is obtained, melting at about 75° C., whilst in the presence of African copaiba oil, which is rich in cadinene, the m.-pt. of the mixed hydrochlorides is raised to about 85° C. by 5 per cent.; about 111° C. by 10 per cent.; and about 114° to 115° C. by 20 per cent. In testing Para copaiba oil for gurjun oil by Turner's reaction 3 drops of the sample are dissolved in 3 c.c. of glacial acetic acid, and the solution treated with 2 drops of freshly prepared 10 per cent. sodium nitrite solution, and then cautiously with concentrated sulphuric acid. In the presence of gurjun oil the acetic acid solution becomes dark violet within thirty minutes. Since certain pure copaiba balsams give a faint positive reaction in this test, the author modifies the method by using 1 drop of the sample and 1 per cent. sodium nitrite solution, and only taking note of colorations produced within five minutes. When a positive result is obtained by this method, 170 grms. of the sample are separated by distillation into three fractions of 50 grms. each up to 145° C. Each of these fractions is dissolved in 300 grms. of acetone (containing water), and the solution oxidised by adding, with constant shaking, 160 grms. of powdered potassium permanganate in portions of about 3 grms. every fifteen minutes. After oxidation is complete, the acetone solution is decanted, and the manganese residue shaken, first with 200 grms. of acetone, and then with 200 grms. of ether. The solvent is evaporated, and the residual oil dried and fractionated under a pressure of 2 to 3 mm., so as to obtain three fractions, boiling respectively below 130° C., 140° C., and 165° C. The second and, especially, the third fractions are then subjected to the semi-carbazone test as follows:

Two grms. of the fraction are added, drop by drop, to a concentrated aqueous solution of 1·1 grm. of semi-carbazide hydrochloride, to which has been added 1 grm. of potassium acetate dissolved in 3 grms. of alcohol and 1 drop of acetic acid, and the final mixture is cleared by the addition of sufficient alcohol. The semi-carbazone is collected after two days, washed with water, dried on porcelain, and weighed. Its m.-pt. ranges from 215° to 228° C., rising to 234° C. after recrystallisation from boiling alcohol. The high specific rotation (+317°) in concentrated aqueous chloral hydrate solution is characteristic of the semi-carbazone from gurjun oil.

C. A. M.

Estimation of Hydrocarbons in Beeswax and Carnauba Wax. A. Leys. (*J. Pharm. Chim.*, 1912, **5**, 577-591.)—A method of estimating hydrocarbons in waxes is based upon the fact that the higher alcohols in beeswax, etc., are soluble in a mixture in equal parts of amyl alcohol and concentrated hydrochloric acid, whilst hydrocarbons may be separated as a solid cake from the solvent. Ten grms. of the wax are saponified with 25 c.c. of alcoholic potassium hydroxide (45 grms. per litre of absolute alcohol) and 50 c.c. of crystallisable benzene in a flask, with a tubulus and tap at the side, so that it may also serve as a separating funnel. After heating the mixture for twenty minutes, 50 c.c. of hot water are added, the boiling continued under a reflux condenser for another ten minutes, and the flame then withdrawn. The liquid soon separates into two layers, and the lower soap solution is then drawn off without cooling. The residual benzene is heated again for ten minutes with 50 c.c. of water, which is subsequently drawn off as before, and the benzene is then evaporated, leaving a residue of alcohols and hydrocarbons. This is treated little by little with 100 c.c. of hot amyl alcohol, and the whole afterwards mixed with 100 c.c. of concentrated hydrochloric acid, and gently heated, with continual stirring, until the mixture has become completely fluid. The flask is now slowly cooled, and the cake of hydrocarbons at the top is separated from the semi-crystalline mass below, redissolved in the 50 c.c. of the mixture of amyl alcohol and hydrochloric acid, separated as before, pressed between filter-paper, heated on the water-bath in a tared basin, and weighed. The wax alcohols in the solvent are separated by heating the liquid with an excess of water, cooling the mixture, drawing off the lower layer of diluted hydrochloric acid, evaporating the amyl alcohol, taking up the residue with benzene, and evaporating the solution in a tared basin. As a further test the liquid fatty acids of the wax may be estimated by treating the lead salts with benzene. Two samples of pure beeswax thus examined gave the following results: Alcohols, 39·21 and 39·60 (melting at 77° C.); hydrocarbons, 10·44 and 13·03; and liquid fatty acids, 8·40 and 8·52 per cent. respectively. In the presence of ceresin or other foreign hydrocarbons, the amount of beeswax is calculated from the ester value, and the corresponding quantity of hydrocarbons deducted from the total hydrocarbons separated. Carnauba wax thus examined yielded 49·22 per cent. of alcohols (m.-pt. 81° C.), but no hydrocarbons.

C. A. M.

Refractive Index of Beeswax. L. Feldstein. (*Chem. Engineer*, 1912, **15**, 211-212.)—To obtain data as to the refractive index of pure beeswax at temperatures above the m.-pt., twenty-seven samples of known purity were examined. These gave results falling within the limits of 1·4398 and 1·4451 at 75° C., whilst samples known to contain small quantities of foreign substances gave values outside these limits. Since all pure waxes give a sharp reading at 75° C., it is recommended that that temperature should be adopted for the estimation, and that the calculation of results to the corresponding values at 40° C. should be abandoned. C. A. M.

Detection of Benzoic Acid in Foods. O. Biernath. (*Veröffentl. a. d. Gebiete d. Militärsanitätswesens*, 1912, Heft 25; through *Chem. Zentralbl.*, 1912, I., 1929.)—The reaction of A. Jonescu (*J. Pharm. Chim.*, 1909, **29**, 523) with 1 drop of 1 per cent. ferric chloride solution and 3 or more drops of 1 per cent. hydrogen peroxide solution is very sensitive; it renders possible the detection of 0·001 grm. benzoic acid in foods, by distillation, in fifteen minutes. Mineral acids, volatile fatty acids, and other volatile acids hinder the reaction. If the first distillate from material containing benzoic acid does not yield a reaction within fifteen minutes, four subsequent distillates from the residue will each give the reaction within fifteen minutes. Salicylic acid, accompanying the benzoic acid, may be destroyed by means of alkaline permanganate in the liquid obtained by distilling the sample with 20 c.c. of water and 0·5 c.c. of sulphuric acid. The benzoic acid is then distilled off, and its presence determined as before. O. E. M.

Analytical Constants of Some Vegetable Fats. H. Sprinkmeyer and A. Diedrichs. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 581-596.)—The following results were obtained with samples of different vegetable fats which are now being used for edible and other purposes:

	$[N]_{D\,40^\circ}$ C.	Iodine Value.	Saponification Value.	Acid Value.	Melting-Point ° C.	Solidifying Point ° C.	Reichert-Meissl Value.	Polenske Value.	Unsaponifiable Matter.
Mowrah butter—									
Crude, 23 samples:									Per Cent.
Lowest	50·7	61·2	190·4	15·0	24·5	16·6	1·70	0·40	1·82
Highest... ...	53·2	64·2	192·7	58·1	28·4	22·2	—	—	—
Refined, 9 samples:									
Lowest	52·8	60·2	190·0	1·3	26·4	18·7	—	—	—
Highest...	54·2	63·8	192·0	3·5	28·4	19·6	—	—	—
Shea butter—									
13 samples:									
Lowest	58·2	54·4	177·0	9·0	43·4	21·0	1·40	0·45	3.60
Highest... ...	60·7	60·0	188·0	66·6	45·2	25·2	—	—	10·01
Adjab fat—									
Crude, 1 sample:	52·4	59·0	182·8	129·0	42·5	39·3	2·50	0·50	7·40
Stillingia tallow—									
Refined, 3 samples:									
Lowest	46·7	39·5	200·9	0·6	41·3	26·7	0·20	0·50	0·30
Highest... ...	47·1	40·5	202·4	0·9	42·5	27·5	0·80	0·60	0·49
Borneo tallow—									
Refined, 1 sample	46·7	29·7	196·6	1·0	28·7	22·6	—	—	0·50
Tulucuna fat—									
Refined, 1 sample	51·7	64·9	194·8	—	37·5	32·3	2·30	0·50	—
Dika fat—									
1 sample	36·5	2·9	242·0	4·0	33·0	—	0·22	5·50	—
Malukang butter—									
1 sample	44·2	49·4	253·0	11·4	—	—	45·50	0·60	—

Mowrah butter is obtained from the seeds of *Bassia longifolia*, adjab fat from the seeds of *Mimusops djave*, shea butter from the seed kernels of *Bassia Parkii*, tulucuna fat from the seeds of *Carapa procera*, dika fat from the seeds of *Irvingia gabonensis*, and malukang butter from the seeds of *Polygala butyracea*. With the exception of tulucuna fat, and possibly stillingia tallow, all the above-mentioned fats are suitable for edible purposes. Tulucuna fat possesses a bitter taste. W. P. S.

Behaviour of Milk when treated with Ammonium Sulphate, and a New Method for the Estimation of Lactose. E. Salkowski. (*Zeitsch. physiol. Chem.*, 1912, **87**, 89-95.)—When milk is saturated, or nearly so, with ammonium sulphate, the proteins are precipitated completely together with the fat, and a clear solution is obtained in which the lactose may be estimated polarimetrically. Fifty c.c. of the milk are shaken with 17·5 grms. of ammonium sulphate, and, when the salt has dissolved, the mixture is diluted to 100 c.c. with saturated ammonium sulphate solution. The whole is shaken and passed through a dry filter, the filtrate being examined polarimetrically. W. P. S.

Detection of Saponin in Beverages and Foods by Hæmolysis. C. Sormani. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 561-566.)—A résumé is given of the papers published by A. Rusconi (*Boll. Soc. Med. Chir.*, Pavia, 1910, 1911, 1912) on the detection of saponin by hæmolysis—that is, the property possessed by certain substances of dissolving the red colouring matter from blood-corpuscles when the latter are suspended in a suitable medium. The blood-corpuscles are obtained by submitting defibrinated blood to centrifugal action with 1 per cent. sodium chloride solution, separating the corpuscles, and suspending them in physiological salt solution. The liquid or substance to be tested must not contain alcohol or carbon dioxide, and must be neutral in reaction; before being added to the corpuscle liquid it is rendered isotonic or slightly hypertonic by the addition of 2 per cent. of sodium citrate. The test may be used for the detection of *Agrostemma githago* in flour, the injurious properties of these seeds being due to the presence of saponin. In this case the flour is made into a dough with 2 per cent. citrate solution, and the dough is then kneaded with a further quantity of the citrate solution, the extract obtained being filtered, and a portion of the filtrate used for the test. The mixture of corpuscle liquid and the solution to be tested is placed in a thermostat for three hours, at the end of which time the red colouring matter will have been dissolved, and the supernatant liquid will appear coloured, should hæmolysis due to the presence of saponin have taken place. Red wine contains a substance which inhibits hæmolysis by saponin, but which may be removed by precipitation with albumin. The test will detect the presence of 1 part of saponin in 10,000 parts of beer, or in 40,000 parts of aerated water. W. P. S.

Detection of Saponin. J. Rühle. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912 **23**, 566-577.)—The author has investigated various tests for the detection of saponin, and finds that Vamvakas' reaction, and colour reactions with sulphuric acid, with Frohde's reagent, and with naphthol, thymol, and ox-gall, are all unreliable, chiefly owing to the presence of interfering substances in the articles under examination (*cf.* ANALYST, 1912, 22). Saponin may, however, be readily detected, even in the presence of glycyrrhizin, by means of its hæmolytic properties. The action of the saponin is dependent on the amount of cholesterol contained in the corpuscles, those containing the largest quantity being most resistant to the hæmolytic action of the saponin. If cholesterol be added to the corpuscle liquid, a point is reached finally at which saponin has no action, and in this way saponin may be differentiated from other hæmolytic substances. In carrying out the test, 1 c.c. of a corpuscle liquid (obtained by mixing 1 c.c. of defibrinated blood with 99 c.c. of a 0·9 per cent. sodium chloride solution, submitting the mixture to centrifugal action, pouring off the supernatant liquid, and diluting the residue to 100 c.c. with the sodium chloride solution) is mixed in a test-tube with from 1 to 3 c.c. of the solution to be tested. If, after the lapse of some length of time, the corpuscles have dissolved, a further quantity of the liquid under examination is mixed with cholesterol and the test repeated; the absence of hæmolytic action in this test indicates definitely that saponin is present. About 4 parts of cholesterol will inhibit the action of 20 parts of saponin. The cholesterol is most conveniently added to the saponin solution in the form of an ethereal solution, the mixture being then heated to 36° C. for some hours in order to expel the ether. When only very small quantities of saponin are being dealt with, the test may be carried out on a microscope slide, the hæmolytic action of the saponin being observed under the microscope. W. P. S.

Polarimetric Estimation of Starch. C. J. Lintner. (*Zeitsch. angew. Chem.*, 1912, **25**, 1177.)—Wenglein's method of hydrolysing the starch with sulphuric acid of sp. gr. 1·7 is liable to give irregular results in hot weather owing to the hydrolysis proceeding too far. The substitution of hydrochloric acid for sulphuric acid is not practicable, since, even without heating, the stage of gelatinisation followed by colloidal solution may be rapidly passed. Under the conditions found most suitable for obtaining constant results, barley starch gave $[\alpha]_D = 198^\circ$ either with sulphuric acid of sp. gr. 1·7 kept cool, or with acid of sp. gr. 1·4 at 22° C., the latter being found the more reliable. The method finally based on these experiments was as follows: 2½ grms. of the finely-ground barley are thoroughly mixed with 5 c.c. of 96 per cent. (by vol.) alcohol in a 100 c.c.-flask with a wide neck, and 50 c.c. of sulphuric acid of sp. gr. 1·4 (=50 per cent. of sulphuric acid) introduced with continual shaking. The flask is then left for an hour, with frequent shaking, at 22° C., after which 10 c.c. of a 2 per cent. phosphotungstic acid solution are added, and the contents made up to 100 c.c. with sulphuric acid of sp. gr. 1·3 (=40 per cent. acid). After thorough shaking, the mixture is filtered, and examined polarimetrically, using sodium light. The rotation multiplied by 10·1 gives the amount of starch in the air-dried barley. C. A. M.

New Method for the Estimation of Glycerol in Wines. C. Beis. (*Bull. Soc. Chim.*, 1912, **11**, [iv.], 618.)—Existing methods for the estimation of glycerol in wines usually involve removal of the sugar by means of barium or calcium hydroxide, variations in the quantities of which lead to inconsistent results; too little allows sugar to pass into the glycerol, while too much causes the retention of glycerol. To meet this difficulty the following method was devised: 100 c.c. of ordinary wine, 50 c.c. of sweet wine, or 25 c.c. of wine over 1·035 sp. gr., are neutralised in a porcelain basin with barium hydroxide and concentrated at 70° C. to a syrup, mixed with sand, and extracted by warming with acetone below its boiling-point. The first portion of acetone is sufficient in quantity to prevent its dilution by the water in the residue below 95 per cent. of acetone; succeeding portions are smaller. (40 to 50 c.c.) Each portion is cooled and filtered; the process is carried on until the volume of the filtrate reaches 200 c.c. Two aliquot parts of the filtrate are evaporated without ebullition. The sugar is estimated by Fehling's solution in one of them. To the other is added five times its volume of water, and powdered barium hydroxide; a few milligrams in excess of the weight of sugar, if this is not more than 0·050 grm.: a weight equal to that of the sugar if this is between 0·050 and 0·300 grm.; and four-fifths of the weight of the sugar if between 0·300 and 0·500 grm. Sand is added, and the mixture extracted with 40 c.c. of acetone, followed by several portions of 25 c.c. Each extract is filtered, and the united filtrate evaporated in a suitable vessel and the residual glycerol weighed. O. E. M.

Analyses of Wines from Eastern France. G. Filaudeau. (*Ann. Falsific.*, 1912, **5**, 296-303.)—The wines examined comprised samples from the departments of Meurthe-et-Moselle (10 samples), Meuse (9), Vosges (8), Haute-Saône and Doubs (10), and Jura (10). The results obtained were normal as compared with those found in other years for these wines. W. P. S.

Analyses of Wines from the Department of Gironde. E. Dubourg. (*Ann. Falsific.*, 1912, **5**, 281-296.)—Results of analyses of 206 samples of red wines and 71 samples of white wines from different districts in the department of Gironde (France) are recorded, the wines all being of the 1911 vintage. The average percentage results for all the red wines were: Alcohol, 9·8; total solids, 23·58; ash, 2·42; reducing sugars, 3·75; volatile acid, 0·85; non-volatile acid, 2·70; potassium hydrogen tartrate (from the total potassium), 4·60; (from the tartaric acid), 1·95. In the case of the white wines (excluding the sauternes) the average results were: alcohol, 10·1; total solids, 28·47; ash, 1·74; reducing sugars, 7·74; volatile acid, 0·55; non-volatile acid, 3·19; potassium hydrogen tartrate (from the total potassium), 2·69; (from the tartaric acid), 2·20. As compared with other years, the red wines contained less non-volatile acid and a correspondingly larger amount of potassium. These irregularities were less accentuated in the case of the white wines. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Small Quantities of Bilirubin. E. Herzfeld. (*Zeitsch. physiol. Chem.*, 1912, **77**, 280-284.) — When an alcoholic solution of bilirubin is treated with a few drops of a solution of *p*-dimethylaminobenzaldehyde (prepared by dissolving 20 grms. of this substance in 500 c.c. of hydrochloric acid and adding 500 c.c. of water) a green coloration is produced, the intensity of which is proportional to the amount of bilirubin present, and this amount may be estimated by examining the mixture with the spectrophotometer. For the estimation of bilirubin in blood, 20 c.c. of the latter are mixed in a mortar with absolute alcohol, then transferred to a 100 c.c. flask, diluted to about 75 c.c. with alcohol, and 2 c.c. of the reagent are added. The mixture is heated on a water-bath until the green coloration has developed, then cooled, and diluted with alcohol to the mark. The solution is now examined with the spectrophotometer. The quantity of bilirubin, x, is calculated from the extinction coefficient, e, observed, according to the formulæ:

$$x = \frac{0\cdot00132 . e}{e}.$$

W. P. S.

Mechanism of Coagulation. J. Duclaux. (*Comptes rend.*, 1912, **154**, 1426-1429.)—A theory of coagulation by osmotic action is based upon the following hypotheses: (1) The osmotic pressure of a solution of several substances is the sum of the partial osmotic pressures; and (2) in a state of equilibrium the relative proportions of different substances that a membrane will allow to pass will be the same on each side of the membrane, the only differences being in the proportions of substances that cannot pass through the membrane. If, for example, to alcohol containing one substance in solution a second soluble substance be added, the alteration in the osmotic pressure will depend upon the size of the molecule of the added substance. If the molecules are very small, the increase of volume caused by their addition will be negligible; whereas, if they are large, there will be considerable increase in volume and a corresponding decrease in the pressure. If, further, two vessels, A and B, are charged with the same alcoholic solution, and separated by a membrane porous to water and alcohol, and a substance capable of reducing the osmotic pressure in B and of not traversing the membrane, be added, water will pass from B into A. But since, according to the second hypothesis, the relative proportions of water and alcohol must always be the same in A and B to obtain equilibrium, alcohol must also pass from B into A in such quantity that the composition of the liquid in A remains constant in so far as concerns the relative proportions of water and alcohol. But under such conditions the osmotic pressure in B must remain lower than in A, and consequently the osmosis will only cease when the whole of the original liquid in B has passed through the membrane into A. The added substance will separate spontaneously. If the original solution contained one grm.-molecule per litre, it is necessary that the grm.-molecule of the added substance should occupy in solution a volume greater than 1 litre before such conditions of osmosis are brought about; and this is usually the case with colloidal substances. In colloidal solutions the place of the porous membrane is assumed to be taken by the free space between the particles of the colloidal substance, and this space will be indefinitely enlarged by the force of osmotic action, until separation of the colloid occurs spontaneously.

C. A. M.

Studies on Enzyme Action. II. Hydrolytic Action of Some Amino-Acids and Polypeptides on Certain Esters. K. G. Falk and J. M. Nelson. (*J. Amer. Chem. Soc.*, 1912, **34**, 828-845.)—The authors suggest as a tentative hypothesis that the hydrolytic action of lipase is due to an optically active substance of protein character, readily hydrolysed in aqueous solution to form lipolytically inactive substances. The characteristic groups of protein substances are the amino and carboxyl groups, free or combined. The possible hydrolytic actions of some simple amino-acids and polypeptides were therefore studied by observing the quantity of acid produced at 38° C., when methyl acetate, ethyl butyrate, and olive oil were dissolved or suspended in aqueous solutions containing glycin, alanin, phenylalanin, leucin, glycylglycin, leucylglycin, glycylleucin, diglycylglycin, aspartic acid, and glutamic acid. Three sets of measurements were necessary for each experiment: with solutions containing amino-acid or polypeptide alone, ester alone, and with a mixture containing both. Sörensen's formol method (ANALYST, 1908, **33**, 19) was used for the estimation of amino-acids and polypeptides, except that $\frac{N}{10}$ soda was used for the titrations in place of $\frac{N}{5}$; phenolphthaleïn was used as indicator throughout, most of the end-points being a distinct pink colour, and not the definite red recommended by Sörensen. Many hundreds of determinations are tabulated, and there was found to be a satisfactory agreement between the results obtained and those already published by Sörensen, in so far as the work covered the same ground. Glycin and alanin showed the greatest amount of action with ethyl butyrate, and least with methyl acetate. Phenylalanin showed a markedly greater action with methyl acetate, less with ethyl butyrate, and least with olive oil. Leucin gave practically no action with the three esters. Glycylglycin gave the same slight action with methyl acetate and ethyl butyrate, but none with olive oil. With leucylglycin, glycylleucin, and diglycylglycin, maximum, though small, actions were obtained with ethyl butyrate, very slight but distinct with olive oil, while with glycylleucin and diglycylglycin and methyl acetate, negative values were obtained. With aspartic and glutamic acids the order of magnitude of action was methyl acetate, methyl butyrate, olive oil, considerably greater action being caused by the former, as was to be expected from the greater ionisation constant of the former. The selective action with different esters is strongly suggestive of the selective action of lipases from different sources with different esters. It seems probable that many of these selective actions of the lipases may be reproduced with amino-acids and polypeptides of varying structure or in the presence of other substances. There is no evidence, however, that the hydrolytic action of lipase is to be attributed to amino-acids or polypeptides. The specific groupings present in the amino-acids or polypeptides which show this activity may be present in more complex substances, such as the proteins, and from this point of view the study of the hydrolytic actions of the decomposition products, such as the amino-acids from preparations possessing lipolytic activity, and of the more complex polypeptides or other substances synthesised from them, may throw light upon the substances capable of causing such lipolytic action. H. F. E. H.

Kumagawa-Suto Method of Estimating Fat in Animal Substances.

R. Watanabe. (*Biochem. Zeitsch.*, 1912, **41**, 71-77.)—As the result of a critical examination of this method (ANALYST, 1908, **33**, 362), the author finds that it is quite reliable for the direct estimation of fat in the following animal substances without drying the latter before saponification: Flesh, heart, liver, pancreas, kidneys, intestines, bones, skin, ascitic and pleural fluids, and frogs. In the case of blood, defibrinated blood, blood-plasma, blood-serum, and brains, better results are obtained when the substances are extracted with alcohol, and the extract used for the estimation of the fat (*cf.* ANALYST, 1910, **35**, 527). Suprarenal glands yielded, after saponification, 27 per cent. of matter soluble in petroleum spirit, but more than one-half of this consisted of unsaponifiable substances.

W. P. S.

Loss of Fat during the Drying of Animal Tissues. M. Tamura. (*Biochem. Zeitsch.*, 1912, **41**, 78-101.)—Shimidzu has already drawn attention to the fact that an apparent loss of fat takes place when animal tissues are dried before the fat is estimated by the Kumagawa-Suto process (*cf.* ANALYST, 1910, **35**, 527), and this is confirmed by the result of an extended investigation carried out by the author. If, however, the substance be dried as quickly as possible in quantities of from 100 to 200 grms., with the addition of an equal quantity of alcohol, the loss of fat due to oxidation does not usually exceed 0·5 per cent. of the total quantity of fat present.

W. P. S.

Blue Colour-Reaction of Phosphotungstic Acid with Uric Acid and Other Substances. O. Folin and B. Macallum. (*J. Biol. Chem.* 1912, **11**, 265; through *Chem. Zentralb.*, 1912, I, 1928.)—Phosphotungstic acid in presence of saturated sodium carbonate solution gives a blue coloration not only with uric acid, but also with phenol, tyrosin, tannic acid, thymol, orcinol, resorcinol, vanillin, and phloroglucinol. The reaction appears to be characteristic of aromatic compounds containing a hydroxyl-group in the *p*-position. Moreover, it appears to be due to an impurity in the phosphotungstic acid, and not to the acid itself. O. E. M.

Refractive Indices of Solutions of Certain Proteins. VI. The Proteins of Ox Serum: a New Optical Method for the Estimation of the Various Proteins in Ox Serum. T. B. Robertson. (*J. Biol. Chem.* 1912, **11**, 179; through *Chem. Zentralbl.* 1912, I, 1929.)—The alteration *a* in the refractive index of a solvent produced by dissolving in it 1 grm. of protein was the same, whether the proteins were dissolved in the native serum, or whether they were precipitated with alcohol, washed with alcohol and ether, dried, and dissolved in $\frac{N}{100}$ potassium hydroxide solution. The value of *a* was also independent of the dilution, and was not altered when the serum was made acid. For the proteins of ox serum the value of *a* is 0·00195. The refractive power of the mixed proteins in ox serum represents the sum of the refractive powers of the individual proteins. For refractometric purposes the non-proteins of the serum may be regarded as a $\frac{1}{8}$-grm. molecular sodium chloride solution. The value of *a* for the albumins of ox serum dissolved in $\frac{3}{8}$ saturated or weaker ammonium sulphate solution is identical with the value for solutions in distilled water: it is 0·00177. The discovery of the additive character of the refractive indices rendered it possible to modify the method of Reiss so that the individual proteins hitherto identified in blood serum could be estimated separately. Ox serum was found to contain 2·34 per cent. soluble globulins, 0·76 insoluble globulins, and 5·4 per cent. total albumins. O. E. M.

Estimation of Sugar in Blood and Urine. E. Frank. (*Zeitsch. physiol. Chem.*, 1912, 78, 165-166.)—The author replies to criticisms of his method for the estimation of sugar in blood (ANALYST, 1910, **35**, 260). W. P. S.

Estimation of Benzoic Acid, Hippuric Acid, and Phenaceturic Acid in Urine. H. Steenbock. (*J. Biol. Chem.*, 1912, **11**, 201; through *Chem. Zentralbl.*, 1912, I, 1930.)—For the estimation of hippuric acid 100 c.c. of urine are boiled for two hours with 10 grms. sodium hydroxide and 25 c.c. of hydrogen peroxide; the hippuric acid is thus converted into benzoic acid, and the colouring matter of the urine is oxidised. The liquid is rendered acid with sulphuric acid, bromine water is added and the liquid made up to 200 c.c. and filtered; 50 c.c. of the filtrate are extracted with ether, the ether evaporated off, and the residue sublimed and weighed. To estimate the phenaceturic acid, the sublimate is titrated with $\frac{N}{20}$ sodium hydrioxide, and the proportion of the benzoic acid to the phenylacetic acid formed from the phenaceturic acid calculated from this titration and the weight of the dry sublimate. O. E. M.

New Method for the Estimation of Hippuric Acid in Urine. O. Folin and F. F. Flanders. (*J. Biol. Chem.*, 1912, **11**, 257; through *Chem. Zentralbl*, 1912, I, 1930.)—A mixture of 100 c.c. urine and 10 c.c. of 5 per cent. sodium hydroxide solution is evaporated to dryness on the water-bath; the hippuric acid in the urine is thus converted into benzoic acid. The residue is transferred by means of 25 c.c. of water and 25 c.c. of concentrated nitric acid to a flask fitted with a reflux condenser, and boiled for four and a half hours with 0·2 grm. copper nitrate to destroy colouring matter and similar substances in the urine. The contents of the flask are saturated with ammonium sulphate in a separating funnel, and extracted with chloroform, and the united chloroform extracts washed once by shaking with a solution of sodium chloride containing hydrochloric acid, and the benzoic acid in the chloroform extract titrated with $\frac{N}{10}$ sodium ethoxide and phenolphthaleïn. O. E. M.

Estimation of Phenol in Urine. M. Hensel. (*Zeitsch. physiol. Chem.*, 1912, **78**, 371-381.)—The method proposed depends on the fact that phenol is dissolved from its acid aqueous solution by ether, and that it can be extracted from the ethereal solution by means of sodium hydroxide, but not by sodium hydrogen carbonate. Five hundred c.c. of the urine are rendered alkaline and evaporated to 100 c.c., the solution being then acidified with phosphoric acid and distilled with frequent additions of water, until all the phenol has passed over, as is shown by the distillate ceasing to give a reaction with Millon's reagent. The distillate is then extracted four successive times with ether, the united ethereal extracts are shaken four times with a 4 per cent. sodium hydrogen carbonate solution, and afterwards the same number of times with $\frac{N}{1}$ sodium hydroxide solution. The sodium hydroxide extracts are united, acid added until the alkalinity of the solution is equivalent to

20 c.c. of $\frac{N}{10}$ solution, and the phenol in the alkaline solution is titrated by Penny and Kossler's method. The method yields trustworthy results in the case of urines containing large quantities of carbohydrates. A method described by Mooser (*Zeitsch. physiol. Chem.*, 1908, **63**, 155), in which the distillate from the urine, acidified with phosphoric acid, is redistilled from calcium carbonate in a current of carbon dioxide, and the phenol titrated in this distillate, was found to yield accurate results.

W. P. S.

Estimation of Urea in Urine by Riegler's Method. A Correction Factor. T. Ekecrantz and S. Erickson. (*Zeitsch. physiol. Chem.*, 1912, **79**, 171-176.)—In the estimation of urea in urine by Riegler's method (*c.f.* ANALYST, 1912, 100), substances other than urea, such as uric acid, purine bases, creatinine, neucleo-albumin, etc., are decomposed and yield carbon dioxide and nitrogen, and the results are, consequently, too high. Those substances which interfere may be removed by treating the urine with twice its volume of a solution containing 10 per cent. of phosphotungstic acid and 1 per cent. of hydrochloric acid of sp. gr. 1·12, and removing the precipitate by filtration after the lapse of four hours; the urea is then estimated in the filtrate. In most cases, however, sufficiently accurate results for all practical purposes may be obtained by treating the urine directly and multiplying the result obtained by 0·926 in order to correct for the amount of nitrogen liberated by the substances other than urea.

W. P. S.

Detection of Savin Oil in Toxicological Cases. J. Hamalainen. (*Biochem. Zeitsch.*, 1912, **41**, 241-246.)—Savin oil contains about 50 per cent. of sabinol acetate, which constitutes the toxic principle of the oil; this acetate on hydrolysis yields sabinol, which, in the body, combines in part with glycuronic acid, and is excreted in the urine as sabinol-glycuronic acid. When the latter is boiled with a mineral acid a cymene is formed, and the identification of the latter has been suggested as a means of detecting savin oil in urine. Other oils, however, such as turpentine, thuja oil, etc., when administered to animals, pass into the urine in the form of derivatives which yield cymene. Sabinol-glycuronic acid may be identified by means of the characteristic crystalline salts it yields with certain bases, particularly with strychnine, and the following method is described by the author for the detection of savin oil in cases of suspected poisoning by this substance: The urine is treated with neutral lead acetate in neutral or feebly acid solution, the precipitate formed is separated by filtration and washed, and, after the filtrate has been rendered ammoniacal, basic lead acetate is added as long as a precipitate is produced. The basic lead salt is collected on a filter, washed with water, decomposed with cold 5 per cent. sulphuric acid, the lead sulphate is separated, and the solution neutralised with barium carbonate. After filtration, the filtrate is concentrated, preferably under reduced pressure, and the barium is precipitated by treating the warm solution with hot strychnine sulphate solution. The mixture is filtered while hot, and the filtrate allowed to cool. Strychnine sabinol-glycuronate separates out in the form of brilliant needles which melt at 196° C. with decomposition. This salt has 2 molecules of water of crystallisation; it is fairly soluble in hot water and but slightly soluble in cold water, alcohol, ether, acetone, chloroform, benzene, petroleum spirit, or ethyl acetate. On concentrating the mother-liquor, a strychnine salt with 3 molecules of water crystallises out, melting at 193° C. It is more readily soluble than the first-mentioned salt, and on cooling its solution rapidly, the salt with 2 molecules of water is obtained. Urine from rabbits, which had received a dose each of 1·5 c.c. of savin oil, yielded crystals of strychnine sabinol-glycuronate when examined by the above method. W. P. S.

Microchemical Reaction of Spermatic Fluid. P. Aleixandre. (*Zeitsch. anal. Chem.*, 1912, **51**, 473-475.)—A reaction proposed by De Dominicis (*Resveglio Medico*, May 15, 1910) consists in treating a trace of the fluid with a drop of a saturated gold tribromide solution on a microscope slide, heating the mixture to boiling, and examining it under the microscope. At a magnification of 300 diameters rectangular or cross-shaped crystals, having a garnet colour, are observed. The crystals are soluble in alcohol and in alkali solutions, but not in acids. The author has examined this test, and finds that it is not very sensitive; the crystals are rarely obtained unless the spermatic fluid is of a concentration of more than 2 per cent. Quadratic crystals are also frequently obtained, and are due to the presence of cholin.

W. P. S.

Colorimetric Method for the Estimation of Uric Acid. E. Riegler. (*Zeitsch. anal. Chem.*, 1912, **51**, 466-470.)—The method depends on the blue coloration which is obtained when uric acid is treated with phosphomolybdic acid and disodium phosphate. The coloration is not given by albumin, peptones, albumoses, creatine, creatinine, or sugar, although these substances yield a similar colour when treated with phosphomolybdic acid and potassium or sodium hydroxides. For the estimation of uric acid in urine, 1 c.c. of the sample is placed in a test-tube, and in two similar tubes are placed, respectively, 1 c.c. of a 0·1 per cent. uric acid solution and 1·2 c.c. of the same urine from which the uric acid has been removed by means of ammonium chloride. (This is effected by heating 10 c.c. of the urine with 3 grms. of ammonium chloride to 40° C., and filtering the mixture after the lapse of thirty minutes; 1·2 c.c. of the filtrate is employed, in order to allow for the increase of volume due to the ammonium chloride.) To each of the tubes are then added 2 c.c. of a 10 per cent. phosphomolybdic acid solution, and a sufficient quantity of a 5 per cent. disodium phosphate solution to make the contents of each tube up to 10 c.c.; a mark may be provided on the tubes for this purpose. After the contents of the tubes have been mixed, they are heated over a flame until small bubbles of gas are evolved, then cooled, and the colorations compared in a colorimeter. The coloration yielded by the urine is first compared with that given by the known quantity of uric acid. The apparent quantity of uric acid in the 1·2 c.c. of urine from which the uric acid has been removed is also estimated by comparison with the standard, and its quantity deducted from the amount found in the urine itself. This correction has to be made, as urine contains substances other than uric acid, which give a coloration with the reagents. Should the urine contain albumin, this must be separated by boiling the urine and filtering it before the estimation is carried out. The standard solution of uric acid is prepared by boiling 0·1 grm. of uric acid and 0·1 grm. of sodium hydrogen carbonate with 50 c.c. of water, cooling the solution, and diluting it with water to a volume of 100 c.c. W. P. S.

ORGANIC ANALYSIS.

Angeli's Reaction for Aldehydes. O. Baudisch and J. H. Coert. (*Ber.*, 1912, **45**, 1775-1779.)—Angeli's test for aldehydes is based on the fact that an aqueous solution of the sodium salt of nitrohydroxylaminic acid forms with various aliphatic and aromatic aldehydes hydroxamic acids which are readily detected in minute quantities by their characteristic iron reaction. The sodium salt of nitrohydroxylaminic acid first splits up into nitrosyl-sodium and sodium nitrite:

$$NaO.N : NO.ONa = NONa + NaNO_2.$$

The nitrosyl-sodium, or perhaps free nitrosyl, combines with the aldehyde; the hydroxamic acid produced may be isolated in the form of its internal complex copper salt. Form-hydroxamic acid may also be produced by the action of light on an aqueous formaldehyde or methyl alcoholic solution of potassium nitrate or nitrite, owing to the formation of nitrosyl or nitrosyl-potassium. In this reaction it appears probable that the combination of nitrosyl with formaldehyde or methyl alcohol takes place through intermediate stages, forming first nitroso-methyl alcohol, which then changes to methylene-nitronic acid, and, finally, to form-hyrdoxamic acid. If this course be actually followed, the formation of the nitroso alcoholate should be accompanied by the temporary appearance of a blue-green coloration. In performing Angeli's test in the ordinary way no such coloration can be observed; the change is too rapid. But in presence of certain substances—*e.g.*, methyl acetate, acetone, ethyl acetate, or gelatin—Angeli's salt shows with aldehydes the blue-green colour of the intermediate nitroso compound. Angeli's salt is dissolved in a little water with a large excess of methyl acetate, and the mixture is shaken; aqueous formaldehyde is added, the liquid being rotated; the methyl acetate then acquires a blue-green colour, which lasts for several seconds. J. F. B.

Precipitant for Amino-Acids. C. Neuberg and J. Kerb. (*Biochem. Zeitsch.*, 1912, **40**, 498-512.)—When a dilute solution of an amino-acid (for instance, glycin) is rendered distinctly alkaline with sodium carbonate, and then treated with a 25 per cent. mercuric acetate solution, a white precipitate is obtained, and, on the addition of from 4 to 5 volumes of alcohol, the amino-acid is precipitated almost completely. The following results were obtained with various amino-acids, the figures representing the percentage of the total nitrogen precipitated: Glycin, 95·7; *d*, *l*-alanin, 95·8; *d*-alanin, 97·3: *d*, *l*-valin, 70·0; leucin, 96·6; *l*-asparagin, 98·6; *d*-glutaminic acid, 97·2; *d*, *l*-serin, 94·9; *l*-cystin, 98·1; *d*, *l*-phenylalanin, 97·9; *l*-tyrosin, 96·9; *d*, *l*-prolin, 75·4; *l*-histidin, 99·4; *l*-tryptophan, 97·7; *d*-glucosamine, 99·5; α-β-diaminopropionic acid, 99·4; *d*, *l*-isoserin, 97·8; δ-amino-valeric acid, 96·1. W. P. S.

Detection and Estimation of Arsenic in Organic Compounds. G. Bressanin. (*Gazz. Chim. Ital.*, 1912, **42**, 451-455.)—The method of estimating arsenic by means of the insolubility of its iodide in sulphuric acid may also be used with organic compounds. Treated with potassium iodide in sulphuric acid of sp. gr. 1·45, precipitates of different kinds were obtained with commercial organic compounds. Thus atoxyl gave a maroon precipitate; sodium cacodylate, a vinous red; and arrhenal, a yellow precipitate. The precipitations were not complete, and Ehrlich's "606" (di-hydroxy-diamino-arseno-benzene hydrochloride) gave no precipitate in acid of this concentration. For the estimation of arsenic in such compounds it is necessary to decompose them completely by heating 0·2 to 0·4 grm. with 10 c.c. of sulphuric acid over a very minute flame (to prevent liberation of cacodylic oxide), after which the arsenic may be quantitatively precipitated as described before (*loc. cit.*). Ehrlich's "606" is sold as a canary-yellow powder, decomposing at 175° C. without melting. It dissolves in sulphuric acid of sp. gr. 1·84 with decomposition, and the solution, when diluted to sp. gr. 1·45, now gives a precipitate with potassium iodide. Silver nitrate gives a yellow coloration, followed by a gelatinous precipitate; copper salts give a blue precipitate; picric acid a yellow precipitate; and potassium ferrocyanide a green precipitate, emitting hydrocyanic acid on heating. A positive reaction is obtained with Gutzeit's test, and Fehling's solution is reduced on heating. C. A. M.

Esterification of Monamino Acids by Means of Ethyl Iodide. Separation of Pyrrolidonecarboxylic Acid from Glutamic Acid. E. Abderhalden and K. Kautzsch. (*Zeitsch. physiol. Chem.*, 1912, **78**, 115-127.)—The silver salt of pyrrolidonecarboxylic acid is readily esterified when boiled for two hours under a reflux condenser with an excess of ethyl iodide. After the silver iodide has been removed by filtration, the ester may be obtained in the form of a white crystalline mass by evaporating the solution under reduced pressure over sulphuric acid; it melts at 60° to 61·5° C. Under similar conditions, glutamic acid, aspartic acid, asparagin, and prolin remain unaltered. When a mixture of the silver salts of pyrrolidonecarboxylic acid and glutamic acid is boiled with ethyl iodide, the former is esterified, and may be separated from the insoluble silver salt of glutamic acid by means of benzene. Attempts to isolate pyrrolidonecarboxylic acid in the digestion products of casein resulted in the formation of an ester which could not be identified; this ester yielded glutamic acid hydrochloride when hydrolysed with hydrochloric acid. W. P. S.

Influence of Metallic Carbonates on the Estimation of the Volatile Substances in Coal. E. Prost and M. Ubaghs. (*Bull. Soc. Chim. Belg.*, 1912, **26**, 216-222.)—Estimation of the volatile substances in coal may give an erroneous idea of the calorific value of the coal or its suitability for gas manufacture, unless account be taken of the amount of carbon dioxide in the volatile substances. In the case of two samples of coal more than 2·5 per cent. of carbon dioxide was present in the volatile substances, whilst other samples gave amounts ranging from 0·5 to 2 per cent. The gas was found to originate from carbonates of iron, calcium and magnesium in the coal. This was found to be the explanation why a coal yielding 15 to 16 per cent. of volatile substances was suitable for the manufacture of coke, whilst another coal from the same source, yielding more than 17 per cent. of volatile substances, was quite unsuitable for the purpose. C. A. M.

Combustion of Volatile Organic Liquids. L. Clarke. (*J. Amer. Chem. Soc.*, 1912, **34**, 746-747.)—A combustion tube is drawn out at one end, and a tube bearing a female ground glass joint is sealed on. That part of the tube which is within the heating zone of the furnace is filled with copper oxide, held in place by plugs as usual, and the end remote from the ground glass joint is provided with a perforated rubber stopper for attachment to the usual sulphuric acid tube and potash bulb. The end of the tube bearing the ground glass joint protrudes about 4 inches from the end of the furnace. The liquid to be analysed is weighed out in a small stoppered U-tube of the type in which the stoppers close the limbs, and, by turning, may be made to put the side tubes in or out of connection with the U. To one side tube is sealed a male ground glass joint, which has been ground to fit the ground seat on the combustion tube. The stoppers of the U-tube and the joint between U-tube and combustion-tube are very lightly greased with vaseline for the upper third or fourth of their ground surfaces. The unlubricated space below, wherever pervious, is filled with air, forming an effective cushion to prevent absorption by the vaseline of any vapour, should this be soluble in paraffin. After weighing, the U-tube is connected to the hot combustion-tube, the other arm being connected to the usual apparatus for drying the air on its way to the combustion-tube. The stopcocks are turned, and the stream of air allowed to pass over the surface of the liquid, carrying forward vapour to the combustion-tube. If the liquid has a boiling-point as low as that of ether, it is well to immerse the lower end of the U-tube in cold water to prevent too rapid volatilisation. For liquids boiling at 100° to 125° C., it is best to immerse the lower part of the U-tube in water at 50° to 60° C. With acetone and similar liquids, neither heating nor cooling is necessary. G. C. J.

Estimation of Caoutchouc as Tetrabromide. Utz. (*Gummi Zeit.*, 1912, **26**, 968-970; through *Chem. Zentralbl.*, 1912, I., 1797-1798.)—In order to avoid the loss of bromine which various authors have found to take place when caoutchouc tetrabromide, prepared by Budde's method, is heated with nitric acid in presence of silver nitrate, the author recommends the method of Baubigny and Chavanne for the estimation of the bromine. The caoutchouc tetrabromide is decomposed by means of chromic and sulphuric acid mixture; a slight modification in the apparatus, consisting in the introduction of a stoppered dropping funnel, is advised. The author, independently of Hübener, has devised a method of bromination in aqueous solution: 0·1 grm. of caoutchouc is treated in a Lintner's pressure-flask with 60 to 70 c.c. of water and 5 to 7 c.c. of bromine, and heated in the closed flask for some hours in the water-bath at 50° to 60° C. When the bromination is complete, the liquid is poured off through a filter, and the contents of the flask are extracted with boiling water. The residue of caoutchouc bromide is washed with alcohol and ether and digested with chloroform for six hours at the ordinary temperature. The bromide is reprecipitated by an excess of petroleum spirit, and, after settling, is collected on a filter, washed with alcohol and ether, dried *in vacuo* at the ordinary temperature, and the bromine estimated in Baubigny and Chavanne's apparatus. The bromine found ranged from 70·12 to 70·25 per cent. in six estimations; theoretical, 70·14 per cent. for $C_{10}H_{16}Br_4$. A sample of Para rubber showed 93·96 and 93·72 per cent. of caoutchouc by this method. The author is investigating the error due to the presence of nitrogenous impurities in the raw rubber. J. F. B.

Latent Heats of Vaporisation of Mixed Liquids. Part III.: Mixtures of Associated with Non-associated Liquids, and the Detection of Solvates in Mixtures of Liquids. D. Tyrer. (*J. Chem. Soc.*, 1912, **101**, 1104-1113.)—Results are given for the boiling-points, vapour compositions, latent heats at constant pressure, and latent heats at constant composition for the following mixtures: Benzene and ethyl alcohol, chloroform and methyl alcohol, chloroform and acetone, carbon tetrachloride and ethyl alcohol. It was found that the mixture of chloroform and acetone gives a maximum boiling-point; the three other mixtures give minimum boiling-points. From a study of the latent heats of such mixtures a method is established by which the existence of solvates in the mixtures can be detected. In all four cases studied solvates are formed. Molecular association in liquids is the result of the combination of hydroxyl groups, and when two hydroxyl substances are mixed solvates are always formed. When a hydroxyl substance is mixed with one containing no such group, the latent heat affords a reliable test for the formation of solvates. In normal mixtures each constituent retains its own latent heat unaffected by the presence of the other constituent; thus the latent heat of either constituent can be calculated from the latent heat of the mixture and the latent heat of the other constituent. Applying this rule to the case of an associated liquid in admixture with a non-associated liquid, the apparent latent heat of the associated constituent can be calculated at various concentrations. If no solvates are formed, the latent heat of the associated liquid in the mixture should gradually diminish as the concentration gradually diminishes, owing to the progressive dissociation of the associated molecules, until at infinite dilution it becomes constant, indicating complete dissociation. If this is not the case, then undoubtedly to some extent solvates are formed in the mixture. In the mixtures above mentioned the diminution was not steady, but the latent heat in all cases, over a portion of the range of concentrations, increased with increase of the dilution. This means that solvates are formed, but it is not possible to determine their amount or composition. The acetone apparently functions in the above case as an hydroxyl substance. J. F. B.

Estimation of Ferrocyanides. H. E. Williams. (*J. Soc. Chem. Ind.*, 1912, **31**, 468-471.)—The following process is stated to be free from most of the possible sources of error inherent in the Feld method: Half a gram of the ferrocyanide is dissolved in 100 c.c. of water, and the liquid distilled after adding 0·05 to 0·1 grm. of cuprous chloride (dissolved in a few drops of hydrochloric acid, or a little saturated sodium or potassium chloride solution) and 25 to 30 c.c. of 4N sulphuric acid. The distillate is passed from the condenser into absorption-flasks containing caustic alkali solution. The ferrocyanide is completely decomposed, and the hydrocyanic acid distilled, by about half an hour's gentle boiling. The cyanide in the alkaline hydroxide solution is then titrated in the usual way with standard silver nitrate solution, in the presence of a few drops of potassium iodide solution. Much larger quantities of ferrocyanide may be taken if desired, and in such cases it is only necessary to add an amount of cuprous chloride equal to 2 to 3 per cent. of the weight of ferrocyanide. Sufficient sulphuric acid must be present to give an excess of acid over that required to form ferrous sulphate and sodium bisulphate. The cuprous chloride used should first be freed from cupric salt by washing with a little dilute sulphuric acid. The addition of cuprous chloride decomposes the ferrocyanide, forming cuprous cyanide, which is decomposed by the acid present, yielding hydrocyanic acid and cuprous chloride again, which latter then attacks a further quantity of ferrocyanide. Both soluble and insoluble ferrocyanides may be determined by the method, but if the substance be dry it should be finely powdered. Ferric ferrocyanide requires more cuprous chloride than above stated for its complete decomposition.

For the estimation of the ferrocyanide content of cyanogen mud or spent oxide, a weighed quantity of the dried material, from which the sulphur has been removed by extraction with carbon disulphide, is boiled with sodium hydroxide, filtered, washed to a definite volume, and a portion of the liquid distilled with cuprous chloride and sulphuric acid as before. The results on spent oxides are higher, generally by about 0·2 to 0·4 per cent., than those given by the titration method, and are more accurate. Thiocyanates are stated to have no influence on the distillation; if present in spent oxide, a larger amount of cuprous chloride should be added to insure their precipitation before distilling (*cf.* Colman, ANALYST, 1910, **35**, 295). A. R. T.

Direct Estimation of Geraniol in Citronella Oil. J. Dupont and L. Labaune. (*Ann. Chim. anal.*, 1912, **17**, 210-213.)—The method depends upon the conversion of the citronellal into a nitrile, which is not affected by the potassium hydroxide in the estimation of the geraniol as geranyl acetate. Ten grms. of citronella oil (or of a mixture of geraniol and citronellal) are shaken for two hours at the ordinary temperature with an aqueous solution of hydroxylamine, prepared by dissolving 10 grms. of hydroxylamine hydrochloride in 25 c.c. of water, adding a solution of 12 grms. of potassium carbonate in 25 c.c. of water, and filtering the mixture. The oil rising to the surface is decanted, dried over anhydrous sodium sulphate, and boiled under a reflux condenser for one and a half hours with twice its volume of acetic anhydride. Under these conditions the citronellal oxime is converted into citronello nitrile. After washing, neutralisation, and drying, 2 grms. of the acetylated oil are saponified with $\frac{N}{2}$ alcoholic potassium hydroxide solution, and the amount of geraniol calculated from the result in the usual way. The difference between the total products of acetylation in a preliminary test and the amount of geraniol thus found, gives the quantity of citronellal. Thus, a Ceylon oil containing 60·2 per cent. of acetylisable products was found to contain 43 per cent. of geraniol, whilst a Java oil contained 43 per cent. of geraniol and 40 per cent. of citronellal. The results given by test mixtures were in close agreement with theory. C. A. M.

Oil of the Southern Cypress. A. F. Odell. (*J. Amer. Chem. Soc.*, 1912, **34**, 824-826.)—The cones of the Southern cypress (*Taxodium distichum*) contain considerable quantities of a volatile oil, consisting of *d*-pinene, 85 per cent.; *d*-limonene, 5 per cent.; a pseudo terpene alcohol (? sabinol), 2 per cent.; carvone, 3 per cent.; tricyclic sesquiterpene, 3 per cent.; the remainder being composed of substances boiling above 275° C. No aldehydes were found in the oil. H. F. E. H.

Estimation of Hydrazine. G. S. Jamieson. (*Chem. News*, 1912, **105**, 268.) —According to the author, Rimini's method requires much time, while the following procedure, based on the general method of Andrews (ANALYST, 1903, **28**, 306), is stated to be rapid and accurate: Hydrazine salts, or the double salts of hydrazine and the metals, are analysed by titration with a standard solution of potassium iodate (3·567 grms. KIO_3 per litre) in presence of hydrochloric acid and chloroform. Suitable proportions are 20 c.c. water, 30 c.c. hydrochloric acid, and 6 c.c. chloroform. The solution of potassium iodate is run in gradually, the reaction proceeding according to the equation:

$$N_2H_4 \cdot H_2SO_4 + KIO_3 + 2HCl = N_2 + ICl + 3H_2O + KCl + H_2SO_4.$$

The liquid is shaken between each addition of iodate, until the chloroform, which at first is coloured, becomes decolorised. Metallic double salts (such as zinc and hydrazine sulphate) may be examined by this method, and the published results are accurate. In the case of the nickel double salt, which is only sparingly soluble, the reaction proceeds very slowly. A. R. T.

Detection and Estimation of Methyl Alcohol. F. Wirthle. (*Chem. Zeit.*, 1912, **36**, 700.)—In testing spirits for methyl alcohol, the first 50 c.c. (at most) of the alcoholic distillate from 100 c.c. are distilled and twice fractionated, so as to obtain first 15 c.c. and finally 3 c.c. as the final fraction. One c.c. of this distillate is oxidised by the official potassium permanganate method, after the addition of 4 c.c. of dilute (1 : 2) sulphuric acid, the liquid filtered, and 1 c.c. of the colourless filtrate heated with 5 c.c. of concentrated sulphuric acid. On treating the cold mixture with 5 mgrms. of solid morphine hydrochloride, a reddish-violet coloration is produced in the presence of as little as 20 mgrms. of methyl alcohol (corresponding to about 5 per cent of the denatured alcohol in the spirit). At the same time a control test is made with a mixture of 20 mgrms. of methyl alcohol and 1 c.c. of 90 per cent. ethyl alcohol. If the preliminary test has given a strong violet coloration, and the spirit contains about 40 per cent. (by volume) of alcohol, 60 c.c. (from 100 c.c.) are taken, whilst with a higher alcoholic strength a proportionately smaller quantity of the distillate is used. The initial temperature of distillation in a fractional distillation-flask affords information as to the amount of methyl alcohol present. Thus a liquid containing 10 per cent. of methyl alcohol begins to distil at 75° C., and after five minutes the temperature will have risen to 76·8° C., whilst in the presence of 20 per cent. of methyl alcohol the initial temperature will be 71·5° C., rising, after five minutes, to 75·5° C. It is advisable to treat the residue from the first distillation with alcohol and to fractionally distil it again, the process being repeated so that from solutions containing about 10 per cent. of methyl alcohol about 80 c.c. in all are obtained, from 20 per cent. solutions about 100 c.c., and from 30 per cent. solutions about 125 c.c. The mixture of methyl and ethyl alcohols is now converted into iodides by heating it under a reflux condenser with iodine and amorphous phosphorus; these are separated by fractional distillation, and the methyl iodide identified by its saponification value (394·3). For the conversion of 10 c.c. of methyl alcohol into methyl iodide 31·7 grms. of iodine and 1·79 grm. of amorphous phosphorus are required, whilst ethyl alcohol requires only 22 grms. of iodine and 1·79 grm. of phosphorus for the formation of the corresponding compound. The necessary amounts may thus be approximately calculated from the temperatures observed in the fractional distillation, a slight excess of iodine being taken. The iodides from a quantity of distillate corresponding to 10 c.c. of alcohol are distilled into 10 c.c. of water, decolorised by shaking with 14 to 16 c.c. of 10 per cent. potassium hydroxide solution, separated from the supernatant liquid, washed twice with water, allowed to stand overnight, and then run into a tared flask, which is immediately closed and weighed. The small residue of iodides remaining in the burette is collected in a narrow calibrated tube, and its weight calculated from the specific gravity. Since 10 c.c. of methyl alcohol yield 31·35 grms. of methyl iodide, and 10 c.c. of ethyl alcohol yield 24·0 grms. of ethyl iodide, each 0·736 grm. of iodide in excess of 24 grms. corresponds to 1 c.c. of methyl alcohol. To obtain reliable quantitative results it is advisable to dilute the distillate from the spirit under examination with sufficient 96 per cent. ethyl alcohol to give a mixture containing approximately 10 per cent. of methyl alcohol, whilst at the same time a blank test is made with a mixture of methyl and ethyl alcohols containing 10 per cent. of the former. C. A. M.

Activity of Organic Nitrogen as measured by the Alkaline Permanganate Method. C. H. Jones. (*J. Ind. Eng. Chem.*, 1912, 4, 438-441.)—It is generally recognised that soluble organic nitrogen is readily available as plant-food, whereas nitrogen in insoluble combinations may or may not be so available. Thus, though dried blood and hoof meal contain most of their nitrogen in insoluble forms, they are known to possess high manurial value, whereas there is reason to believe that the nitrogen of peat, roasted leather, garbage tankage, and some other materials, is not immediately available as plant-food. For this reason certain States of the American Union forbid the use of these latter materials in compound fertilisers offered for sale. In other States, agricultural chemists are called upon to report on the manurial value of mixed fertilisers which may contain nitrates, ammonia, and soluble and insoluble organic nitrogenous substances. The valuation of the insoluble nitrogen has hitherto presented a difficulty for the reasons stated, and the author now proposes to differentiate between the insoluble nitrogen which is converted into ammonia by digestion under standard conditions with alkaline permanganate and that which is not so transformed. That which is converted into ammonia under the conditions described in the paper is designated active insoluble organic nitrogen, and it is suggested that this active nitrogen should be assigned a manurial value but little inferior to the soluble nitrogen, whereas the residue of insoluble nitrogen, designated inactive, is held to have a very low value indeed. These views find support in field experiments—referred to but not described in the paper—and in the fact that in dried blood, high grade tankage, fish, hoof meal, cotton-seed meal, and many other substances which give manurial results in close relation to their nitrogen contents, the proportion of inactive nitrogen, as determined by the author's method, is low, whereas the same method shows that the inactive insoluble organic form is the predominant form of nitrogen in peat, garbage tankage, charred leather, and other substances of notable nitrogen content but negligible manurial value.

Active insoluble organic nitrogen is determined as follows: Total insoluble nitrogen is first determined by extracting 2 grms. of the sample on a filter-paper, with water at room temperature, until the filtrate amounts to 250 c.c. The filter-paper and its contents are subjected to the Kjeldahl process. So much of the substance is now weighed out as contains 50 mgrms. of insoluble nitrogen, and, if a mixed fertiliser, is extracted with water, as in the determination of total insoluble nitrogen. Highly nitrogenous raw materials are mixed in a mortar with 2 grms. of powdered rock phosphate before extracting with water, and very oily materials should have a preliminary treatment with ether. The filter and insoluble matter are dried at a temperature not exceeding 80° C., and the insoluble matter is then transferred to a 500 to 600 c.c. round-bottomed flask, together with 20 c.c. of water, a few glass beads, and 100 c.c. of an alkaline permanganate solution containing 150 grms. of caustic soda and 25 grms. of potassium permanganate per litre. The flask is connected to a condenser as for an ammonia determination, the receiver being charged with a measured quantity of standard acid. The contents of the flask are digested short of boiling for at least thirty minutes, local overheating of the insoluble residue being avoided by the use of wire gauze and asbestos-paper between the flame and the flask. About 95 c.c. are now distilled, preferably at such a rate that the operation requires an hour. A tendency to froth may be overcome by prolonging the digestion until this no longer manifests itself, and then distilling at a somewhat brisker rate, so that the whole process occupies about ninety minutes as usual. The ammonia in the distillate is the measure of the active insoluble nitrogen.

The method has been adopted since March, 1911, by the experiment stations of New York, New Jersey, and the New England States, as the result of laboratory and field experiments in 1910. G. C. J.

Action of Permanganate on Organic Substances. J. Hetper. (*Zeitsch. anal. Chem.*, 1912, 51, 409-429.)—In earlier papers (ANALYST, 1911, 36, 232, 422) the author has shown that many organic substances are quantitatively oxidised to carbon dioxide and water by permanganate in acid solution, whilst the members of another large group of compounds may also be estimated by means of permanganate, the reaction being a quantitative one, with acetic acid for one of the end products. The influence of alkaline permanganate has now been studied. In the majority of cases it proves a less useful reagent than an acid solution. For example, ethyl alcohol, which can be quantitatively oxidised to acetic acid in acid solution, is oxidised partly to acetic acid and partly to carbon dioxide in alkaline solution, the former reaction predominating if the action begins in the cold, the latter if the mixture is hot from the start; but at no temperature between 0° and 100° C. can either reaction be suppressed entirely. Methyl alcohol is much more quickly oxidised in alkaline than in acid solution, the products being carbon dioxide and water in either case. Caffeine, theobromine, and antipyrin, which only consume from 40 to 80 per cent. of the theoretical amount of permanganate in acid solution, are almost quantitatively oxidised to carbon dioxide, water (and ammonia) in alkaline solution. Many substances which are oxidised quantitatively in acid solution behave similarly in alkaline solution, but in a much greater number of instances the results in alkaline solution correspond to no single reaction. However, the author thinks that the consumption of permanganate in alkaline solution under standard conditions may prove a useful constant, especially when compared with the corresponding number obtained in acid solution.

The experimental conditions followed in the author's laboratory are as follows: The permanganate solution is $\frac{N}{2}$, and contains 40 gr. of caustic soda per litre. Of this solution, 25 c.c. is placed in a 200 c.c. flask with 65 c.c. of water, and the substance dissolved in 10 c.c. of water. So much of the substance is taken as may be expected to reduce about 10 c.c. of the permanganate, and, if the substance is insoluble in water, it is mixed with 1 grm. of glass powder, not omitting in such a case to add 10 c.c. of water to the contents of the flask to insure uniformity of conditions in all experiments. The flask, with its contents, is heated on the water-bath for one and a half hours; 25 c.c. of $\frac{N}{2}$ oxalic acid (containing 75 c.c. concentrated sulphuric acid per litre) is then added, and the solution titrated with $\frac{N}{2}$ permanganate containing 40 grms. of glacial phosphoric acid per litre. The result is calculated to c.c. of $\frac{N}{1}$ permanganate per gram of substance. The amount of permanganate reduced by forty-two substances under these and some other conditions are given in the paper. G. C. J.

Influence of Peptones on the Estimation of Reducing Sugars by Fehling's Solution. A. Bernardi. (*Biochem. Zeitsch.* 1912, **41**, 160-164.)—Although peptones do not reduce Fehling's solution, their presence in sugar solutions causes more cuprous oxide to be precipitated than is due to the reducing action of the sugar alone. In cases, therefore, where sugar has to be estimated in solutions containing peptones, the latter should be removed by means of phosphotungstic acid previous to the estimation of the sugar. W. P. S.

Physico-Chemical Basis of the Seliwanoff Lævulose Reaction. A. Jolles. (*Biochem. Zeitsch.*, 1912, **41**, 331-332.)—When dextrose is placed in contact with concentrated hydrochloric acid for some months, the solution yields a reaction for lævulose with the Seliwanoff test. The author finds, however, that the polarisation of the sugar is not changed, a fact which shows that either the quantity of lævulose formed is so small that it cannot be estimated, or, which is more probable, that the substance giving the Seliwanoff reaction is not lævulose. The action of dilute alkali solutions on dextrose is much more pronounced, and the change is accompanied by the formation of appreciable quantities of an acid. For instance, 200 c.c. of a 0·5 per cent. dextrose solution were mixed with about 40 c.c. of $\frac{N}{10}$ sodium hydroxide solution; 50 c.c. of this mixture required 9·1 c.c. of $\frac{N}{10}$ acid for neutralisation when titrated at once, but after the lapse of ninety-four hours the same quantity of the mixture used only 7·7 c.c. of the acid, phenolphthaleïn being used as the indicator in both cases. The author also found that the Seliwanoff test would detect the presence of as little as 0·1 per cent. of lævulose in urine containing dextrose.

W. P. S.

Method for Determining the Value of Commercial Starches for Use in Cotton Mills. G. M. MacNider. (*J. Ind. Eng. Chem.*, 1912, **4**, 417-422.)—The value of starch for cotton-mill purposes depends on its property of swelling and forming a viscous solution when treated with hot water, the principal starches used being maize, potato, cassava, and, to a small extent, wheat, sago, and rice. As each of these starches has a different viscosity, to obtain the best results they must each be used in a different manner. No definite relation exists between their behaviour and the size or shape of the grains. The author determines the viscosity of each starch at a high temperature under conditions similar to those in which it is used in the mills, employing for the purpose a Scott viscosimeter in the following manner: 12 grms. of the starch are weighed into a 600 c.c. beaker, 300 c.c. distilled water added (thus making a 4 per cent. solution), and heated over a Bunsen burner, with constant stirring, to the boiling-point and boiled for ten minutes; 200 c.c. of this solution are then poured into a viscosimeter cup, which is jacketed with boiling water, the temperature allowed to become constant, which it does with most starches, at 94° C., and 50 c.c. run out into a graduated vessel, the time being measured with a stop-watch. The number of seconds so required divided by the number of seconds required to deliver 50 c.c. of boiling water (nine seconds) gives the viscosity value.

The recorded results show that not only is there a difference between different starches, but also a considerable variation in the viscosity of different samples of the same starch, only a small part of this latter difference being due to variation in moisture content.

The viscosity of maize starch increases uniformly with the length of time of boiling, and corresponds to the increase in concentration brought about by continued boiling. This is a valuable property of maize starch as compared with others.

Potato starch reaches its maximum viscosity after being boiled five minutes; it then decreases rapidly, the increasing concentration having no effect and after thirty minutes' boiling it has fallen below that of maize starch, which has been boiled for the same length of time.

Cassava starch attains its maximum viscosity at the boiling-point, after which it decreases uniformly with the length of time of boiling, and thus resembles potato starch, though to a less extent; the former starch, therefore, has a much wider application in sizing and finishing.

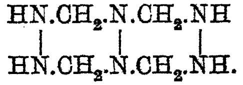
Wheat starch has a much lower viscosity than any of the others examined, and shows a gradual but small increase with the time of boiling. Wheat starch foams more in boiling than any other.

Rice starch, even at the end of thirty minutes' boiling, still shows a value only very slightly greater than unity. The degree to which starches go into solution on continued boiling was examined, and it was noticed that those from roots and tubers developed under ground (potato and cassava) showed a high rate of solution, while those from the grains developed above ground were very insoluble. An examination was made of some "thin boiling starches," which usually consist of maize starch treated in some way to reduce the viscosity. Experiments are described showing the effect of boiling maize starch with borax, alkali, and boric acid, as regards viscosity changes. A small amount of all these reagents reduces, while a slightly larger amount increases, viscosity. H. F. E. H.

Estimation of Thymol, Salicylates, and Allied Compounds. A. Seidell. (*Amer. Chem. J.*, 1912, **47**, 508-526.)—The method given in the author's previous paper on this subject (Analyst, 1909, **34**, 536) is admittedly tedious, and was only suggested as a possible improvement over the ordinary bromate titration in which tribromo-phenol bromide is formed. The control of the too vigorous action of bromine on the compounds under examination has now, in the author's opinion, been accomplished more satisfactorily. The method is as follows: A quantity of 0·1 to 0·5 grm. of thymol or other substance to be estimated is placed in a glass-stoppered bottle, with 1 to 2 c.c. of carbon tetrachloride and 100 c.c. of water. Bromine vapour is poured in till the mixture, after thorough shaking, shows a considerable excess. After half an hour, 5 c.c. of carbon disulphide, immediately followed by 5 c.c. of aqueous potassium iodide (20 per cent.), are added, and the liberated iodine titrated with $\frac{N}{10}$ thiosulphate. An additional amount of potassium iodide is added, and if there is no further liberation of iodine, the burette reading is taken, 5 c.c. or an excess of aqueous potassium iodate solution (2 per cent.) added, and the titration, after thorough shaking, continued till the iodine colour is discharged. The second titration corresponds to the hydrobromic acid formed by the action of bromine on the compound used. Two molecules of hydrobromic acid are equivalent to 1 molecule of thymol; 1 c.c. $\frac{N}{10}$ thiosulphate, therefore, is equal to 0·0075 grm. thymol. The results quoted vary from 99·0 to 100·8 per cent. of the theoretical. Carbon disulphide and tetrachloride serve to remove the compound from the aqueous layer, and the reaction in the organic solvent is more under control, only dibromothymol being produced. Carbon tetrachloride, unlike carbon disulphide, is little acted on by bromine on standing, and is therefore added first; but a satisfactory end-point in the titrations is not obtainable without the addition of the disulphide as described. Salicylic acid may be determined when in admixture with benzoic acid, the latter being unacted on. Other substances tried gave variable results.

A. R. T.

Tetraformal-trisazine from Formaldehyde and Hydrazine Hydrate, a New Reducing Agent for Analytical Use. K. A. Hofmann and D. Storm. (*Ber.*, 1912, **45**, 1725-1730.)—Hydrazine hydrate is employed in analytical chemistry for the precipitation of silver, gold, mercury, platinum, selenium, and tellurium, but its reducing power is too great to permit of fine differentiations among the noble metals, and it also reduces solutions of copper and bismuth. The precipitates are not easily filtered, and firm mirrors are not obtainable on glass. Hydrazine hydrate also attacks glass, and is not free from alkalis. The authors, therefore, recommend as a reducing agent far more under control and free from alkalis and acid, the compound tetraformal-trisazine, $C_4H_{12}N_6$, formed by the combination of formaldehyde with hydrazine hydrate. This substance has the structural formula—



$$\begin{array}{ccccc} HN & .CH_2. & N & .CH_2. & NH \\ | & & | & & | \\ HN & .CH_2. & N & .CH_2. & NH. \end{array}$$

For its preparation, 50 c.c. of 35 per cent. formalin are poured slowly into 50 c.c. of hydrazine hydrate cooled with ice, and after one hour a further 30 c.c. of formalin are similarly added. The mixture is placed in a basin, covered with paper, and left for one or two days, when the yield of crystallised compound should amount to 27 grms. The crystals are filtered off, washed with spirit, redissolved in water, and precipitated with alcohol and ether; they may also be recrystallised as flat, silky needles from slightly alkaline hot water. At 28° C., 100 grms. of water dissolve 14 grms. of the azine. Silver nitrate yields first a white precipitate, changing immediately on warming to a fine adherent metallic mirror; palladium chloride also yields a mirror. Gold chloride yields first a blue colloid, and later a dark precipitate. Copper sulphate gives a reddish-brown solution. The azine is not decomposed by alkalis, from which it crystallises unchanged. In alkaline solution, copper salts are reduced to cuprous oxide, mercury, gold, and silver salts to the metals, whilst platinum and palladium chlorides give stable, reddish-brown solutions. In presence of excess of potassium hydroxide, chromates, molybdates, vanadates, selenites, and tellurites remain unchanged, even at the temperature of the water-bath; but on the addition of ammonium chloride, indigo blue molybdenum oxide, red selenium, or black tellurium are precipitated. Tungstates are not reduced even in ammoniacal solution. Thus tetraformal-trisazine may be recommended as a selective reducing agent which is readily prepared in a perfectly pure form. The substance, though stable towards alkalis, is very sensitive to the action of acids, even carbonic and acetic acids, forming polymerised formalazine, $(CH_2N_2CH_2)_n$, as an insoluble white powder. Ammonia is one of the principal products of the action of hot dilute sulphuric acid. J. F. B.

Examination and Valuation of Turpentine. H. Wolff. (*Farbenzeitung*, 1912, **17**, 1492; through *Chem. Zentralbl.*, 1912, I., 1930.)—The colour is more conveniently determined by a comparison with solutions of iodine, potassium dichromate, etc., than by means of the Lovibond colorimeter. The polymerisation with sulphuric acid, which involves oxidation, should not be omitted (*cf.* Marcusson, *Chem. Zeit.*, 1910, **34**, 285); otherwise, adulteration with residues from the manufacture of turpentine may be overlooked. The distillate obtained before and after the chief fraction in the distillation of turpentine are to be found on the market and are difficult to detect when added to turpentine. The greater part of the residual oil distils earlier when mixed with turpentine than it does alone. On the other hand, with concentrated or fuming sulphuric acid, more separates from the mixture than would be anticipated from the amount of residual oil. It is evident that sulphuric acid has less action in the presence of high-boiling constituents. A separation of more than the normal 2 to 4 per cent. is sometimes shown, when the resinification is well started, even by pure turpentines, in which there is a slight natural increase of these constituents, and, consequently (petroleum being excluded), is not a proof of adulteration with these residues. A small increase in the residue on evaporation, with a large increase in the amount separated by fuming sulphuric acid, points to an adulteration with these waste products, whereas an increase in the resinification is of no value as an indication. A residue of 1 per cent. on evaporation has a noticeably bad effect on the drying-period, whilst 2 or 3 per cent. renders a turpentine useless for many purposes in the varnish and colour industry. O. E. M.

Estimation of Petroleum in Turpentine. H. Wolff. (*Farbenzeitung*, 1912, **17**, 1553; through *Chem. Zentralbl.* 1912, I, 1931.)—The constituents dissolved by nitric acid are, contrary to the statement of Herzfeld (*Chem. Zeit.*, 1910, **34**, 885), cyclic hydrocarbons, since they can be reduced by the slow action of nascent hydrogen, the solution being kept warm. It was found that xylene can be dissolved in concentrated sulphuric acid alone, but that long shaking is necessary; this explains the conflicting results obtained by Marcusson and Herzfeld with turpentines containing xylene. Consequently Herzfeld's method of determining benzene has only a qualitative value, and only then if it is positive and agrees with the other constants (boiling-point, bromine number, etc.). The separation of petroleum or paraffin hydrocarbons by concentrated fuming sulphuric acid is more valuable, especially if, by the method of Utz, the refraction of the separated residue is determined. The Marcusson method is preferable for the exact determination of petroleum and benzene hydrocarbons. In the case of a strongly resinified oil, if there is a considerable separation with concentrated sulphuric acid, even when the Marcusson test gives a negative result, it is better to examine once more the substance separated with sulphuric acid by the Marcusson method. O. E. M

Tests for Determining the Purity of Oil of Turpentine. R. Marcille. (*Ann. Falsific.*, 1912, **5**, 241-251.)—Determinations of the solubility of oil of turpentine in acetic acid, the rise in temperature when mixed with sulphuric acid, and the solubility in sulphuric acid, are stated to yield results which will afford evidence as to the genuineness of a sample of turpentine.

Solubility in Acetic Acid.—The acetic acid employed should contain 98 per cent. of the acid and 2 per cent. of water; it should be capable of dissolving an equal volume of anhydrous carbon disulphide at a temperature of 39 to 40° C., at which point a mixture of equal volumes of the two liquids should just become turbid; if the temperature at which the turbidity is observed is lower or higher than this, the strength of the acid must be adjusted accordingly. Five c.c. of the oil of turpentine are placed in a stoppered graduated tube, and shaken with successive additions of acetic acid until a clear mixture is obtained at a temperature of 15° C. The quantity of the acid required to dissolve 1 part of the oil of turpentine is then calculated. For genuine samples this quantity is usually less than 4. The oil of turpentine may also be distilled, the distillate being collected in five separate portions, which are then submitted to the test. The first fractions dissolve in about 3·5 parts of acetic acid, whilst the fifth fraction may require up to 4·5 parts of acid. The presence of petroleum diminishes the solubility of the oil, the fifth fraction of a sample containing 5 per cent. of petroleum requiring from 6 to 7 volumes of acetic acid. Oil of turpentine adulterated with "benzine" has a greater solubility than pure oil of turpentine.

Thermal Test with Sulphuric Acid.—The method of carrying out this test in the case of fatty oils has been described by Tortelli (ANALYST, 1909, 34, 168). The same procedure is adopted with oil of turpentine, but, as the latter evolves very considerable heat when mixed with sulphuric acid, it is better to perform the test on a mixture of 5 c.c. of the sample and 15 c.c. of "vaseline oil." Genuine oil of turpentine gives a value of from 68 to 72, and the presence of 10 per cent. of mineral oil will diminish this value by about 7.

Solubility in Sulphuric Acid.—One hundred c.c. of petroleum, 20 c.c. of oil of turpentine, and 50 c.c. of sulphuric acid are mixed in a Röse tube, and the increase in the volume of the sulphuric acid is ascertained after the lapse of one hour. In the case of genuine oil of turpentine, the increase will be about 19·2 c.c. (that is, 96 per cent. of the oil is soluble in the acid), whilst with an oil containing 10 per cent. of mineral oil the increase in the volume of the acid will be only 17·1 c.c. The test is useless should "benzine" be present. Care should be taken to examine the petroleum used in the test, as some specimens yield notable quantities of matter soluble in sulphuric acid. W. P. S.

INORGANIC ANALYSIS.

Detection of Small Quantities of Alkali Bicarbonate in Presence of much Normal Carbonate. R. T. Haslam. (*J. Amer. Chem. Soc.*, 1912, 34, 822-823.)—The carbonate suspected to contain bicarbonate is dissolved in recently boiled water, and excess of calcium chloride is added. If alkali bicarbonate was originally present, some calcium bicarbonate will remain in solution with the calcium chloride. After standing five minutes, a portion of the solution is filtered free from calcium carbonate, and a few drops of ammonia are added to the filtrate. An immediate precipitate of calcium carbonate indicates a large amount of bicarbonate in the original sample. With only small amounts of bicarbonate present, the precipitate may not appear for ten minutes. The test will detect 0·1 per cent. of bicarbonate in normal sodium carbonate. The presence of ammonium salts vitiates the results because of the solubility of calcium carbonate in such solutions. G. C. J.

Neutral Ammonium Citrate Solution. A. J. Patten and C. S. Robinson. (*J. Ind. Eng. Chem.*, 1912, 4, 443-446.)—According to the official methods of analysis drawn up for the guidance of American agricultural chemists, the ammonium citrate solution for use in determining available phosphoric acid is to be prepared by dissolving citric acid in water, adding ammonia until the solution is neutral to corallin, and then diluting it with water until the sp. gr. is 1·09. The authors point out that the determination of the end-point with corallin is very difficult, and that purified litmus, though better, is also unsatisfactory as an indicator. Solutions "neutralised" by four experienced analysts were used to extract a phosphatic manure, with the result that the manure was found to contain 2·83, 3·69, 4·17, and 4·82 per cent. of insoluble phosphoric acid. The authors think that the amount of ammonia necessary for neutralisation should be determined by the conductivity method as suggested by

Hall and Bell (*J. Amer. Chem. Soc.*, 1911, 33, 711; *J. Ind. Eng. Chem.*, 1911, 3, 559). The solution is nearly neutralised, and then equal measured portions are transferred to measuring-flasks, and a measured and different amount of dilute ammonia is added to the contents of each flask, so that two flasks at least contain an excess of ammonia and two an excess of acid. The contents of the flasks are then diluted to the mark, and their relative conductivities determined. It is unnecessary to determine the absolute conductivities. Provided the same apparatus is used for all the determinations, the Wheatstone bridge readings may be plotted against the volumes of dilute ammonia, and the break in the curve will indicate the volume of ammonia necessary to neutralise so much of the solution as was taken for the test. The error of this method, even in the hands of persons with no previous experience of conductivity measurements, is only one-tenth the error of the most careful titration with corallin or litmus.

G. C. J.

Separation of Beryllium from Aluminium. M. Wunder and P. Wenger. (*Zeitsch. anal. Chem.*, 1912, 51, 470-473.)—From a mixture of beryllia and alumina, the former may be separated as follows: The mixture is fused with 5 grms. of sodium carbonate in a crucible, which is covered at first, but subsequently uncovered, whilst the mixture is maintained in a state of fusion for two to three hours. The melt is boiled out with water, about a gram of sodium carbonate is added, and boiling continued for a few minutes. Beryllia remains undissolved. It is filtered off, washed, dried, ignited, and weighed, and re-fused with sodium carbonate, and the whole process repeated to insure the solution of the last traces of alumina. In the mixed filtrates, alumina is precipitated by boiling with excess of ammonium nitrate, and is filtered, washed, dried, ignited, and weighed in the usual manner. The worst recorded results are 0·1466 grm. beryllia and 0·1385 grm. alumina found with 0·1472 and 0·1379 grm. respectively present. The results are not stated in such a way as to show what additional error, if any, might be expected to result from the omission of the second fusion.

In practice, more or less iron will, as a rule, be present in the material presented for analysis, and the whole of this iron will remain as insoluble hydroxide with the beryllia, any chromium also present passing as chromate into the solution of sodium carbonate and aluminate. Precipitates of beryllia contaminated by ferric hydroxide are ignited, fused with bisulphate, and the aqueous solution of the melt is poured into a solution of 10 grms. of caustic soda in 150 c.c. of water, and the whole boiled. The precipitate consists of ferric hydroxide free from beryllia, and is washed, re-dissolved in acid, re-precipitated by ammonia, and weighed as ferric oxide as usual. The alkaline solution of beryllia is acidified, and the beryllia precipitated by means of ammonia.

When chromium is present in the original material, it will exist as chromate in the filtrate from the alumina, and may be recovered by reduction with alcohol and precipitation with ammonia.

G. C. J.

Volumetric Estimation of Chloric Acid and Chlorates. A. Kolb. (*Chem. Zeit.*, 1912, 36, 635.)—The high results obtained by the action of hydrochloric acid on chlorate in presence of potassium iodide at the ordinary temperature are due to the presence of atmospheric oxygen, and may be avoided by removing the air from the hydrochloric acid solution by means of a current of carbon dioxide, dissolving the chlorate in water which has been boiled and carrying out the reaction in a vessel filled with carbon dioxide. To 10 c.c. of air-free potassium chlorate solution (about $\frac{N}{10}$) are added 1 grm. of solid potassium iodide and 50 c.c. air-free hydrochloric acid of sp. gr. 1·125 in the order given, and the mixture allowed to stand five to ten minutes in a stoppered vessel in the dark. After addition of 200 to 300 c.c. water, the separated iodine is titrated. If the quantity of chlorate solution is more than 10 c.c., the hydrochloric acid must be increased proportionately, otherwise the reaction is slower. An increased quantity of chlorate in the 10 c.c. is without effect on the reaction-velocity, but more potassium iodide is required to keep the iodine in solution.

O. E. M.

Quantitative Determination of Perchlorates. A. B. Lamb and J. W. Marden. (*J. Amer. Chem. Soc.*, 1912, **34**, 812-817.)—The minus errors which attend estimations of perchlorate by fusion with sodium carbonate or other substances, and determination of the resultant chloride as silver chloride, are shown to be due to loss of chloride by volatilisation. This may be wholly avoided as follows: The substance (0·5 grm.) is weighed into a Jena glass test-tube of about 25 to 30 c.c. capacity. An asbestos plug is pushed down into the tube within 2 inches of the bottom, and a second plug 2 inches nearer the top. The tube is clamped in a nearly horizontal position, and the perchlorate is heated gently until effervescence ceases. A somewhat stronger heat is then applied, until, in from ten to fifteen minutes, the chloride is thoroughly fused. When cool, the contents of the tube, including the plugs, are washed on a Gooch crucible with hot water, and the chloride in the filtrate determined in the usual manner. A correction must be made, if necessary, for chloride in the asbestos used, and in the most exact work 0·1 mgrm. may be added to the weight of silver chloride found, as a correction for the loss with the final wash liquors. Without this last correction, the worst result of eight recorded by the authors is only 0·05 per cent. below the theoretical.

G. C. J.

Estimation of Chromium in Bronzes containing Tin and Antimony. H. Schilling. (*Chem. Zeit.*, 1910, **36**, 697.)—The following rapid modification of von Knorre's method is recommended: Two grms. of the finely-divided turnings are dissolved in 25 c.c. of hot *aqua regia* (1HNO_3 to 4HCl), and the solution mixed with 40 c.c. of sulphuric acid (1:1), and evaporated until white fumes appear. Complete removal of the *aqua regia* is essential. When cold, 200 c.c. of water are added, and the precipitated sulphates dissolved by boiling, with the exception of the lead sulphate. The copper and antimony are now precipitated by means of iron wire in the metallic form, the liquid filtered after five minutes, and the precipitate washed. The filtrate and washings are made up to 500 c.c., heated to boiling, and treated first with a few drops of silver nitrate solution (1 : 20) as a catalytic agent, and then with 10 c.c. of a cold saturated solution of ammonium persulphate. By vigorous boiling the chromium is oxidised to chromic acid, whilst the bulk of persulphate is meanwhile decomposed. The remainder, together with any permanganate that may have been formed, is decomposed by adding 5 c.c. of hydrochloric acid (sp. gr. 1·12) and boiling the liquid for a short time. When completely cold, the chromium is estimated by titration in the usual way. The presence of tin does not affect the accuracy of the results. C. A. M.

Iodimetric Estimation of Copper. K. Sugiura and P. A. Kober. (*J. Amer. Chem. Soc.*, 1912, **34**, 818-822.)—Though the iodimetric method for the estimation of copper is capable of yielding results of a high order of accuracy, this is only the case when the thiosulphate is standardised under conditions very closely resembling those prevailing in actual assays. In consequence of this, many papers have been published on the influence of various amounts of acid, salts, water, etc., the most recent being that of Peters (ANALYST, 1912, 277). Instead of trying to secure uniformity in all the conditions by the methods advocated by Peters and others, the authors think it far more easy to precipitate the copper as hydroxide, to dissolve the washed precipitate in acetic acid, and to titrate the resulting solution with thiosulphate after addition of potassium iodide. One advantage of the method is the stable end-point with starch indicator (no return of the blue colour). The only point in the method requiring close attention is the precipitation as hydroxide. To the solution, containing phenolphthaleïn, 33 per cent. alkali is added drop by drop until a slight precipitate of copper hydroxide remains undissolved. $\frac{N}{5}$ caustic soda free from carbonate (containing baryta) is then added from a burette until a change of colour is observed. Any excess of alkali containing carbonate would hold some copper in solution, whilst a large excess even of caustic alkali would prolong the operation of washing the precipitate. If the precipitation be performed as described, three or four washings suffice. The precipitate with the filter-paper is returned to the flask in which the precipitation was carried out, the precipitate is dissolved in 25 c.c. of 10 per cent. acetic acid, 3 or 4 grms. of potassium iodide are added, and the titration with thiosulphate conducted as usual. The thiosulphate is standardised by precipitating a measured volume of a standard copper solution with subsequent solution of the precipitated hydroxide and titration as described. Obviously, the concentration and nature of the acids and salts in the original solution are without influence on the results, if the washing of the hydroxide be sufficiently prolonged. The amount of washing which the authors give the precipitate cannot remove the whole of the salts, but the test numbers given in the paper show that no sensible error is introduced from this cause. G. C. J.

Estimation of Oxygen and Occluded Gases in Copper. G. L. Heath. (*J. Ind. Eng. Chem.*, 1912, **4**, 402-404.)—The loss in weight on heating copper drillings in hydrogen is not due entirely to oxygen from cuprous oxide and sulphurous acid, but also includes gases derived by the metal from the fuel and refining in the furnace and, in addition, any trace of moisture. In recognition of this fact it is now customary to heat the drillings to constant weight in a current of carbon dioxide before proceeding to the determination of oxygen. The loss in weight during the preliminary heating in carbon dioxide is returned as "absorbed gases," and includes hydrogen, and traces of carbon dioxide, carbon monoxide, and nitrogen. The author finds that the subsequent loss of weight by heating in hydrogen does not express the true oxygen of the cuprous oxide present in the cast metal, for the metal may take up so much hydrogen during the process that the amount of oxygen may be underestimated by more than 20 per cent. An accurate determination of oxygen may be made by passing a current of carbon dioxide through the heated tube for twenty minutes after reduction by hydrogen is complete.

In the author's laboratory the carbon dioxide is purified by treatment successively with a saturated solution of permanganate, solution of silver sulphate, concentrated sulphuric acid, anhydrous calcium chloride, dry chromous chloride, stick phosphorus, phosphoric anhydride, and anhydrous calcium chloride. The hydrogen is purified by means of a 10 per cent. solution of caustic potash saturated with permanganate, concentrated sulphuric acid, heated palladium asbestos, a 5 to 10 per cent. solution of pyrogallol in 50 per cent. caustic potash, and anhydrous calcium chloride. The clean drillings (50 grms.) are placed in a hard-glass tube, 30 cm. long and 6 mm. diameter, with one long bulb or two round ones; and if the copper is very porous or of unknown origin, the sample is dried at 100° C. while passing a current of carbon dioxide, and the tube with its contents re-weighed after cooling and replacing the carbon dioxide by air. Air is once more expelled by carbon dioxide, and this gas continuously passed while the tube is heated to full redness for twenty minutes. After cooling and replacing carbon dioxide by air, the tube is re-weighed, the loss being due to occluded gases in the copper. Hydrogen is now passed through the tube, which at its forward end is connected to a gas-washing device containing 10 c.c. of an ammoniacal solution of cadmium chloride (2 per cent.). According to the fineness of the drillings, from one to two hours at a red heat is required to reduce the cuprous oxide. Finally, hydrogen is expelled from the tube and from the copper by replacing the stream of hydrogen by one of carbon dioxide while maintaining a red heat. After twenty minutes, the tube is cooled by an air blast, the carbon dioxide replaced by air, and the final weighing of the tube made. The loss since the previous weighing represents oxygen and sulphur, and the latter is determined by iodine titration of the contents of the cadmium chloride bulbs after addition of hydrochloric acid and appropriate dilution. Proof of the accuracy of the method is given by a careful electrolytic assay of the reduced drillings. The sum of the copper (and silver) thus found, plus the percentage of arsenic and foreign metals found by complete analysis, should amount to 100 per cent. without any oxygen or occluded gases. In three sets of test numbers given, the totals are 100·0007, 99·9998, and 99·9901. Such check-work implies the use of refined electrolytic methods such as those described by the author (ANALYST, 1911, **36**, 172), which permit of the determination of copper with an error not exceeding 0·001 per cent. (see also ANALYST, 1900, **25**, 253; 1905, **30**, 385).

G. C. J.

Precipitation of the Copper-Arsenic Group and Separation of its Divisions. J. I. D. Hinds. (*J. Amer. Chem. Soc.*, 1912, **34**, 811-812.)—The method of precipitation described has regard to the fact that a fairly high concentration of acid is necessary for the complete precipitation of quinquevalent arsenic, whereas cadmium, antimony, and tin are not completely precipitated unless the acid concentration is kept low. To 50 c.c. of the solution, if approximately neutral, 5 c.c. of concentrated hydrochloric acid and 10 drops of nitric acid are added, or a less amount of hydrochloric acid if the original solution is strongly acid. The solution is concentrated to half bulk, whereby not more than one-thousandth part of the arsenic is lost. The nitric acid serves to oxidise all the tin to the stannic condition, which is desired, because stannous sulphide is not readily soluble in colourless ammonium sulphide; at the same time, some arsenic is oxidised to the quinquevalent condition. Arsenic is now precipitated by passing hydrogen sulphide for ten minutes through the hot solution, which is heated once or twice until it actually boils. About 80 c.c. of water are added, and the current of hydrogen sulphide continued until the liquid is cold, or for fifteen minutes. The precipitate, which contains the sulphides of all the metals of the group, is filtered, washed, and transferred to a beaker, where it is covered with strong ammonia. Hydrogen sulphide is passed through the mixture for two minutes, after which the other sulphides are separated by filtration from those of arsenic, antimony, and tin, which pass quantitatively into the filtrate with only a trace of copper. G. C. J.

Qualitative Analysis without Hydrogen Sulphide. H. Trapp. (*Zeitsch. anal. Chem.*, 1912, **51**, 475-480.)—Higher oxides are first reduced by passing sulphur dioxide through the solution. If insoluble sulphates separate at this stage, they are filtered off and examined separately. After boiling off excess of sulphur dioxide, hydrochloric acid is added, and any precipitate formed is filtered off and examined in the usual manner.. To the solution, or filtrate from insoluble chlorides, nitric acid is added to peroxidise the iron, then ammonia until a precipitate begins to form or until the solution is no longer acid, and finally ammonium sulphide in excess. Any insoluble sulphides are filtered off, and the filtrate is acidified to throw down the sulphides of tin, antimony, and arsenic, if present. These metals, as well as the alkali metals, alkaline earth metals, and magnesium, which pass into the filtrate, are separated and identified in the usual manner.

The ammonium sulphide precipitate, which may contain mercury, lead, bismuth, chromium, aluminium, zinc, iron, manganese, copper, cadmium, cobalt, and nickel is treated with nitric acid, and any undissolved mercuric sulphide is filtered off. Lead is precipitated as sulphate, and bismuth tested for by diluting a portion of the solution. If bismuth is present, it is next separated, after expelling the excess of acid in the solution. On addition of caustic soda and a little bromine and warming, all the remaining metals are precipitated as hydroxides except chromium, aluminium, and zinc, which pass into the filtrate as chromate, aluminate, and zincate, except when iron is also present, when chromium will be found in the precipitate. The filtrate is divided into three portions, in one of which aluminium is precipitated by ammonium chloride; in another, zinc is detected as sulphide, whilst the third is reduced with sulphur dioxide, and any chromium precipitated as hydroxide.

The hydroxides of iron, manganese, copper, cadmium, cobalt, and nickel are digested with ammonium sulphide and washed. From the mixed sulphides iron and manganese are extracted by dilute hydrochloric acid and separated from each other.

by means of sodium acetate. The sulphides of copper, cadmium, cobalt, and nickel, are dissolved in hot concentrated sulphuric acid, and the solution is neutralised with ammonia. Copper and cadmium are precipitated by shaking with zinc dust, the solution of cobalt and nickel is filtered, and these metals precipitated as hydroxides by boiling with excess of caustic soda. The residue of zinc dust with any copper or cadmium is treated with hydrochloric acid, which dissolves the zinc and cadmium, and the latter may be separated from the zinc by boiling the filtrate from the undissolved copper with excess of caustic soda.

An alternative method of analysis, also dispensing with the use of hydrogen sulphide, is described in the paper. G. C. J.

Determination of Iodides by Direct Titration. J. W. Turrentine. (*J. Ind. Eng. Chem.*, 1912, 4, 435-436.)—The method, an empirical one, is intended for the rapid estimation of small amounts of iodide in presence of much chloride, such as is required in a laboratory where large numbers of kelp samples have to be reported on. The solution (10 to 100 c.c.) containing the iodide to be estimated is transferred to a 250 c.c. separator, into which 15 c.c. of dilute (1 : 10) sulphuric acid and 15 c.c. of carbon tetrachloride are also introduced. A solution of permanganate, containing about 1 grm. per litre and standardised in an exactly similar manner on known amounts of iodide, is next added from a burette with shaking after each addition. With the decrease in the concentration of iodide, the solubility of the iodine decreases until, as the end of the titration is approached, the solution appears colourless. The easiest end-point to work to, however, is the persistence for one minute of the pink tint due to excess of permanganate. From the reading, an empirically determined deduction of 0·2 c.c. is made, and the remainder is then the measure of the iodide present. In standardising the permanganate, measured volumes (10 to 100 c.c.) of an exactly 0·1 per cent. solution of potassium iodide are treated in the manner described. By taking varying volumes, not only is the iodine value of the permanganate determined, but also the empirical correction already referred to. A series of numbers is given showing that in the author's hands this correction is about 0·2 c.c. permanganate, and that with this correction the method enables the analyst to determine small quantities of potassium iodide with an error not exceeding 6 per cent. with as little as 10 mgrms. present, nor exceeding 3 per cent. with quantities of 20 to 100 mgrms. The application of the method may be extended by effecting the standardisation in presence of large quantities of any salt, in presence of which the analyst is called upon to determine traces of iodide; but where this other salt is sodium chloride, such procedure is unnecessary. The author frees his carbon tetrachloride from reducing substances by prolonged treatment with iodine, subsequently removing the excess of iodine with thiosulphate and washing the carbon tetrachloride with water. G. C. J.

Potassium Iodide and Mercurous Nitrate as Sensitive Reagents for Tungsten and Molybdenum. E. Kafka. (*Zeitsch. anal. Chem.*, 1912, 51, 482.)—To the neutral solution of tungstate or molybdate, 1 drop of a saturated solution of mercurous nitrate is added, and this is followed by 1 c.c. or more of concentrated hydrochloric acid and excess of potassium iodide. The mixture is shaken vigorously until the green mercurous iodide first formed redissolves. In presence of much tungstate or molybdate, the solution assumes a blue colour at once. With little tungstate or molybdate present, the colour develops more slowly. As little as 0·2 mgrm. of sodium tungstate gives a distinct reaction. To distinguish between tungsten and molybdenum, potassium thiocyanate is added to the blue solution, which will change to blood-red in presence of molybdenum. Solutions containing so little molybdenum as to develop no blue colour in the first reaction may give a distinct red with thiocyanate. The function of the mercurous nitrate in the reaction first described is to increase the reducing power of the hydrogen iodide by forming a stable complex with the liberated iodine. G. C. J.

Separation of Nickel and Palladium by Means of Dimethylglyoxime. W. Wunder and V. Thuringer. (*Ann. Chim. anal.*, 1912, **17**, 201.)—The solution of the two metals is slightly acidified, preferably with hydrochloric acid, and treated with an excess of a 1 per cent. solution of dimethylglyoxime in 2 per cent. hydrochloric acid. After standing for thirty minutes on the hot-water bath the precipitated palladium is collected, washed with boiling water, ignited, and weighed in the metallic form. The filtrate is boiled and rendered slightly alkaline with ammonia, and the precipitated nickel dimethylglyoxime is collected in a Gooch crucible, washed with boiling water and then with 20 per cent. alcohol, and dried at 100° C. until constant in weight. The weight multiplied by the factor 0·2031 gives the amount of nickel. In solutions containing 0·025 grm. of palladium and 0·0846 grm. of nickel, the amounts thus found in six estimations were 0·0248 to 0·0252, and 0·0840 to 0·0843 grm., respectively. C. A. M.

Use of Oxygen under Pressure for Estimating Carbon in Ferro-Alloys. P. Mahler and E. Goutal. (*Compt. rend.*, 1912, **154**, 1702-1705.)—The use of the calorimetric bomb in the estimation of carbon in steel (ANALYST, 1911, **36**, 562) has been extended to the most refractory ferro-alloys. With these alloys, not only must one use litharge as a flux as in the case of very hard steels, but an auxiliary combustible is required. As auxiliary combustible the authors use iron of known carbon content. From 0·5 to 3 grms. of the alloy, according to its supposed carbon content, is taken for combustion, together with two or three times its weight of iron. With alloys very high in carbon, of which only 0·5 grm. can conveniently be taken, the proportion of iron must be increased, as there is some difficulty in effecting complete combustion of charges with a total weight of less than about 3 grms. Of litharge a weight equal to half that of the combustible charge is taken. A second experiment is made under identical conditions, save for the omission of the ferro-alloy. This second experiment provides the correction for the carbon in the soft iron and for any constant errors of the method. The authors now employ caustic soda for absorbing the carbon dioxide, and estimate the amount of the latter by titration before and after combustion, using phenolphthaleïn as an indicator. The test numbers given in the paper show that, when the method does not give results in close agreement with those of the ordinary direct dry combustion method, it gives higher results. In these cases—high-grade ferro-chromes and ferro-silicons—the authors think their higher numbers must be preferred. The following numbers serve to illustrate the difference in results by the two methods. A 69 per cent. ferro-chrome showed 10·6 per cent. carbon by the bomb, and 9·7 per cent. by ordinary direct dry combustion. A 75 per cent. ferro-silicon showed 0·20 per cent. carbon by the bomb, and 0·11 per cent. by the ordinary method. Results by the two methods with ferro-vanadium and ferro-molybdenum agree very closely. G. C. J.

Separation of the Rare Earths. C. James. (*J. Amer. Chem. Soc.*, 1912, **34**, 757-771.)—The paper states in a compendious form the best methods available for the separation of La, Ce, Pr, Nd, Sm, Eu, Gd, Tr, Dy, Ho, E, Tm, Yb, Lu, Ct, Yt, and Sc, in mixtures where all may occur. The methods are suited to large scale operations rather than to analytical practice, one method requiring a few thousand fractional crystallisations. But, though several of these elements can be determined with fair accuracy in minerals or mixtures of oxides in which they are the chief constituents, exact methods for the separation of others are not yet known. Where spectroscopic examination shows one or more of the rarer earths to be present in notable proportion, reference to this paper will show the analyst whether an approximate estimation of these elements is reasonably practical, and, if so, will indicate the necessary steps to be taken. G. C. J.

Improvements in the Ludwig-Sipöcz Method for the Estimation of Water in Silicates. M. Dittrich and W. Eitel. (*Zeitsch. anorg. Chem.*, 1912, **75**, 373-381.)—The method referred to in the title of the paper is that in which the powdered mineral is mixed with dry fusion mixture in a platinum boat, heated in a current of dry air in a combustion tube, and the water absorbed in appropriate drying tubes and weighed. The authors propose to replace fusion mixture by sodium carbonate, chiefly because it is easily obtained anhydrous by three hours' heating below 300° C., and can be mixed with the powdered mineral without risk of the absorption of more than a few tenths of a mgrm. of moisture. They also agree with Hillebrand ("Analysis of Silicate and Carbonate Rocks," p. 87) that the higher melting-point of sodium carbonate makes its use preferable to that of a mixture of sodium and potassium carbonates. The other innovations of the authors relate to the apparatus employed. They use a quartz combustion tube 45 cm. long by 22 mm. diameter, and 0·5 mm. in the wall. The platin-iridium boat, which is 12 cm. long, is provided with a cover and enclosed in a cylinder of thin platin-iridium foil, just fitting the combustion-tube, to protect the latter in case of frothing. The forward end of the combustion-tube is drawn out and connected by a ground joint to the first tared drying-tube. The other end of the combustion-tube is also ground to receive a quartz plug carrying a tube of small diameter, which is connected by rubber tubing to the apparatus for drying the current of air. Sulphuric acid is used for drying the air and for absorbing the moisture given up by the mineral. Heating is effected by a multiple Bunsen burner, supplemented as a rule by five to ten minutes heating with a multiple blowpipe, or an electric furnace may be used with advantage. In the latter case the temperature is raised to 850° to 900° C. in about twenty minutes, maintained at this point for fifteen minutes, and then raised to 1000° to 1050° for ten to fifteen minutes. One grm. of the substance with 6 grms. of sodium carbonate is a suitable charge, and the residue in the boat is immediately available for the determination of silica, alumina, etc. Duplicate determinations of moisture seldom differ by more than a few tenths of a milligram. G. C. J.

Volumetric Estimation of Sulphuric Acid, Nitric Acid, and Nitrous Acid, in Nitrating and Waste Acids. G. Finch. (*Zeitsch. Schiess- u. Sprengstoffwesen*, 1912, 7, 113.)—A wad of cellulose fibre is placed in 50 c.c. of the diluted mixture of acids, excess of barium carbonate added, and stirring continued until the evolution of carbon dioxide nearly ceases, when the liquid is boiled for five minutes. The precipitate of barium carbonate and sulphate is well washed, the filtrate boiled for several minutes with a wad of cellulose and 50 c.c. of $\frac{N}{5}$ sodium carbonate, filtered, the precipitate washed until no reaction is obtained with phenolphthaleïn, and the excess of sodium carbonate in the filtrate titrated back with $\frac{N}{5}$ hydrochloric acid and methyl orange. The cellulose, prepared by steaming ash-free filter-paper cut into small pieces for two days, and kept for use in alcohol, ensures the precipitation of the barium sulphate in an easily filtered condition. The barium carbonate is prepared by precipitating barium chloride solution with sodium carbonate in the cold, and washing thoroughly by decantation.

If $\frac{N}{10}$ barium hydroxide solution is used instead for the neutralisation, the acids may be estimated simultaneously; the total acidity is given by the amount of barium hydroxide used, and the nitric acid, present in the filtrate as barium nitrate, is estimated as before. Nitrous acid, if present, is titrated with permanganate, and deducted from the total acidity. The nitrometer may show a higher total nitrogen than the volumetric method; this is attributed to the presence of lower nitric-acid esters of glycerol, not removed in the after-separation. The error is usually negligible. O. E. M.

Rapid Method of Purifying Sulphuric Acid. G. Bressanin. (*Gazz. Chim. Ital.*, 1912, 42, 456-458.)—The principle of the method of separating arsenic, etc. (ANALYST, 1912, 206), may be applied to the purification of sulphuric acid for toxicological work. The acid is first concentrated to sp. gr. 1·52, then cooled, and treated with 10 c.c. of a 30 per cent. solution of hydriodic acid. After standing for twelve hours it is filtered through glass-wool and purified asbestos, and the iodine eliminated from the filtrate by boiling. Acid containing as little as 0·0005 mgrm. of arsenic per c.c. gives a precipitate with the reagent after some time. The purification must be carried out in vessels of Jena glass, since ordinary glass or porcelain may yield further traces of arsenic to the acid. Tin, copper, cadmium, lead, antimony, and selenium, are also precipitated—the last in elementary form; whilst nitric and nitrous acids are reduced to nitrogen peroxide. Mercury and bismuth are not precipitated, and no precipitate is given by the members of the third and following groups of metals. G. A. M.

New Reagent for Thorium. M. Koss. (*Chem. Zeit.*, 1912, **36**, 686.)—The solution to be tested is rendered strongly acid with hydrochloric acid, and a few drops of a solution of sodium hydrogen hypophosphate ($NaHPO_3$, $2H_2O$) added. With considerable quantities of thorium a white flocculent precipitate forms at once; with small quantities heating or long standing is necessary. Less than 0·0001 grm. of thorium oxide in 1 c.c. of solution may thus be detected. Titanium and zirconium are simultaneously precipitated. Previous treatment with hydrogen peroxide converts the titanium into pertitanic acid, which is not precipitated by the reagent. To remove zirconium, the mixed precipitate, after washing, is oxidised with sulphuric and nitric acids, and the solution precipitated with oxalic acid; zirconium remains in solution. O. E. M.

Quantitative Determination of Yttrium. C. F. Whittemore and C. James. (*J. Amer. Chem. Soc.*, 1912, **34**, 772-774.)—Precipitates of yttria are apt to be contaminated with salts of sodium, potassium, lithium, and magnesium, and with ferric oxide and alumina, if the corresponding elements are present in the solutions from which the yttria is precipitated. Thus yttrium may be overestimated by 5 per cent. if precipitated by caustic soda, ammonia, or oxalic acid, in presence of salts of sodium or potassium. The ammonium salts of several organic acids were tried as precipitant, with unsatisfactory results; but it is found that ammonium sebacate precipitates yttria quantitatively and free from sodium even when large quantities of sodium are present. In presence of potassium, a second precipitation as sebacate is necessary to obtain exact results. In presence of iron, aluminium, magnesium, or lithium, the whole of the yttria may be precipitated free from any trace of these elements by means of oxalic acid, provided the precipitation be made in the cold, and that excess of ammonium chloride is present. G. C. J.

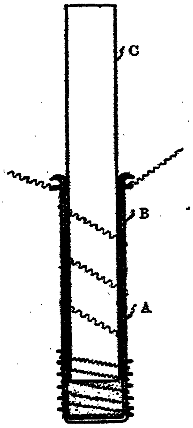
APPARATUS, ETC.

Estimation of the Concentration of Colloidal Solutions with the New Interferometer for Liquids. R. Marc. (*Chem. Zeit.*, 1912, **36**, 537.)—The interferometer described by F. Löwe (*Phys. Zeitsch.*, 1910, **11**, 1047) was sensitive with true solutions to 0·003 per cent., with a 4-cm. cell; it is found to be nearly as sensitive with solutions of dextrin, albumin, and other colloids. The ageing of such solutions on standing was almost without effect on the concentration as determined by the instrument. For small ranges the concentration was found to be nearly enough proportional to the readings to render a calibration curve unnecessary. With colloidal solutions the difficulty of matching the right pairs of lines is increased, and methods for overcoming this are given. As an example of the application of the instrument to concentration determinations in colloidal solutions, adsorption experiments (albumin and dextrin by barium carbonate) are described. The method may be extended to the examination of water: at the waterworks, by examining before and after filtering; for potable waters, before and after shaking with a substance capable of adsorbing colloids, but not affecting substances in true solution. The method is not usually applicable to dyes. O. E. M.

New Apparatus for the Coking Test of Coal. R. Lessing. (*J. Soc. Chem. Ind.*, 1912, 31, 465-467.)—This apparatus is devised to show, more markedly than the usual "crucible method" can, the difference in the coke produced from various coals. The apparatus is also useful for quantitative determinations of the volatile matter, in which respect it gives, generally speaking, results similar to those obtained by the old method. The sources of error of the crucible test are stated to be—

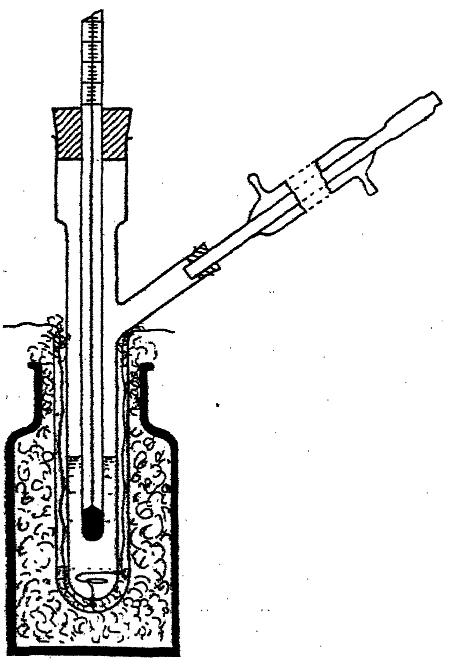
1. *Factors tending to Increase the "Volatile" Value.*—(*a*) Burning of coke in the more or less oxidising atmosphere; (*b*) "spitting" of coal-dust due to the explosive character of gasification of some coals, or rush of currents produced by the flame.

2. *Factors tending to Decrease the "Volatile" Value.*—(*a*) Insufficient coking of the coal; (*b*) secondary catalytic decomposition of volatile products on the walls of the crucible; (*c*) decomposition of volatile products by radiation in the waste space of the crucible; (*d*) deposition of carbon during coking by too rapid primary decomposition of volatile coal substance.



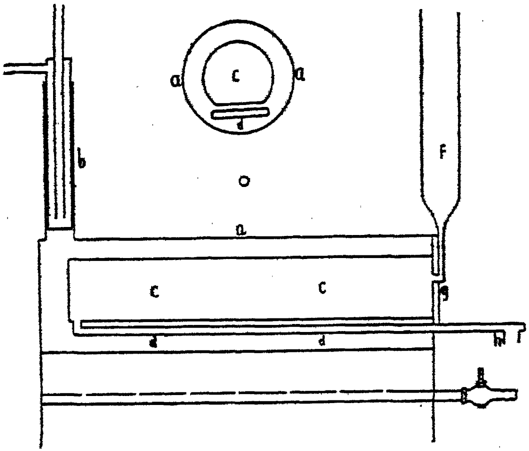
These factors are at work concurrently, and may compensate each other, but their presence explains the difficulty of obtaining agreement under slightly altered working conditions. In order to avoid the drawbacks due to the waste space above the coal, and to the excessive amount of heated surface in contact with the gas and vapours in the crucible test, the author uses a cylindrical vessel, as shown in the figure. *A* is the heating-tube of quartz glass, round which an electric resistance coil of platinum wire is wound. A number of quartz pin-points are fused on this tube to keep the turns of the coil apart. To concentrate the heat where required, the wire is first wound to a coil about 3 mm. in diameter, and this coil is then wound round the heating-tube. The reacting vessel *B* is a flanged quartz tube about 10 mm. in internal diameter, fitting loosely into the heating-tube. (Platinum tubes may be used instead of quartz tubes.) One grm. of the powdered sample of coal is placed in this tube *B*, and a third quartz tube *C*, fitting closely into the reacting chamber *B*, is placed on top of the coal before the commencement of the test. The pressure on the coal may be altered if desired by filling the piston-tube *C* with varying quantities of quartz powder. The outer tube *A* is permanently fixed in an insulating material, such as kieselguhr. Suitable resistance is provided according to the voltage of the supply current, and a rheostat is combined with the furnace itself, so that the temperature can be controlled. When the current is turned on, the different stages of carbonisation can be observed as the temperature rises; moisture goes off quickly, as a rule, some of the occluded gases follow, and, shortly after this, tarry vapours are evolved, increasing in volume according to the coal used for the test, and, finally, the heavier tars and pitchy matter make their appearance, the latter condensing on the cool parts of tube *C*. The test takes from five to seven minutes, and the tubes are then allowed to cool, when the tar at the mouth of *B* may be burnt off without interfering with the coke. The apparatus brings out the peculiarities of cokes from coals, although in the crucible test little or no difference is to be seen. The same kind of coal also gives the same characteristic coke. The paper is illustrated with photographs showing the appearance of the cokes produced by coals in this apparatus, together with their analytical figures. A. R. T.

Modification of the Beckmann Apparatus. E. Knecht and J. P. Batey. (*J. Chem. Soc.*, 1912, **103**, 1189-1193.)—For the determination of the boiling-points of aqueous solutions of certain sparingly soluble dyestuffs, the ordinary types of Beckmann apparatus failed to give satisfactory results owing to superheating effects caused by cohesion of the liquid on the glass walls of the vessel. The authors were therefore led to devise a method of electrical heating by means of a platinum wire in direct contact with the solution. The apparatus (see figure) consists of a tube with a platinum heating coil inside, and somewhat thicker wires fused through the glass for making the connections. When in use the apparatus is supported in an outer cylindrical glass jacket placed in a wide-mouthed bottle, packed at the bottom and round the neck with cotton-wool. With this arrangement the boiling-point of the liquid was found to remain constant to 0·001° C. In employing this method precautions must be taken to avoid any appreciable electrolytic action. The electrolytic effect will depend on the drop in potential across the heating-coil, and when this is reduced below a certain point no electrolysis should take place. In the above apparatus the resistance of the heating-coil at 100° C. was 0·47 ohm; the current used was 6·5 ampères, hence the drop in potential was only 3 volts. In another apparatus the coil had a resistance of only 0·32 ohm. With a current of 7·5 ampères, the drop in potential was 2·2 volts. With a 10 per cent. solution of potassium chloride in this apparatus, no chlorate was formed after boiling for half an hour, and the electrolysis of potassium iodide was very small. Measurements of molecular elevations of boiling-point made in this apparatus were in satisfactory agreement with published results. The authors found the molecular weight of tannic acid in aqueous solution to be between 3000 and 4000. Results have also been obtained for various dyestuffs, including benzopurpurine and indigo white. With the electrical heating constant readings are readily obtained without special precautions.



J. P. B.

Drying Oven. J. H. Coste. (*J. Soc. Chem. Ind.*, 1912, 31, 471.)—This oven can be maintained at a constant temperature, and throughout its area a stream of hot air, or other gas of the desired temperature circulates. As shown in the figure, a horizontal cylindrical copper vessel, *a*, contains a suitable quantity of a pure liquid, the boiling-point of which is near that of the temperature required. This vessel is heated by a series of gas-jets, and the vapour is condensed at *b*, and returned. The muffle-shaped drying-chamber, *cc*, is surrounded by the boiling liquid, or its vapour, with the exception of about one-thirtieth of its external surface (the door). Air which has passed through a flattened tube, *dd*, lying under, but not in contact with, the drying-chamber, enters it at the bottom of the end in the vapour, and leaves through a slit, communicating with the chimney *f*, in the door which is fixed tightly against the end of the chamber by means of steam rubber jointing and a screwed hinging bar against the end of the oven. The area of the drying chamber is small, relative to that of the total heated surface. If desired, the air may be dried before it enters the pre-heater at *h*, or other gases may be used. The drying-chamber will hold six watch-glasses or flat weighing-bottles placed on a copper tray, allowing several determinations of water to be simultaneously carried out, and the substances to be dried rapidly attain a temperature within 1° of that of the vapour. A. R. T.



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REVIEWS.

Allen's Commercial Organic Analysis. Vol. V.: Tannins and the Analysis of Leather. By W. P. Dreaper. 1911. London: J. and A. Churchill. Price 21s. net.

Remembering the painstaking work which distinguished the first edition of this well-known book, the perusal of the sections on Tannins and Leather Analysis has been one of disappointment to the writer. The author has failed to render himself familiar with much recent work, and as a compilation, the chapters must be pronounced uncritical and in places unreliable, and containing much obsolete and useless matter. In several cases work is incorrectly described, and in more than one is attributed to the wrong author.

Such charges are not to be made lightly; and, at the risk of being tedious, they must be supported by detailed instances. On the very first page the tannins as a class are characterised as "notably soluble in ethyl acetate." In fact, their solubility in this reagent is very varied, and in some cases almost nil, and this peculiarity has been used by Stiasny in the separation of different members of the group. Tannins are spoken of as "powerful reducing agents," "especially in alkaline solution," while it is only under the last condition that they show any marked affinity for oxygen. On p. 7 gallotannic acid is mentioned as a glucoside, which is contrary to the modern view. Sumach-tannin is described as "possibly identical with gallotannic acid," while Strauss and Schwendner have shown that this is not the case. Hemlock-tannin is stated to contain phloroglucol, which is incorrect. The tannin of horse-chestnut bark is spoken of as "chestnut tannin," which is misleading, since the very important chestnut extract of commerce is made from the wood of the true Spanish chestnut, *castanea vesca*, which is not even mentioned in the table. The same remark is also to some extent true of "ulmo-tannic acid," since the "ulmo" bark of South America, which is now much used in tanning extract manufacture, is probably not a true *ulmus*.

On p. 8 the Stiasny formaldehyde test for catechol tannins is incorrectly given, and completely misunderstood. The Seyda test with gold chloride on the following page is merely the formation of colloidal gold, and the reaction is produced by any feeble reducing agent. On p. 14 the phloroglucol test with deal-shaving is stated in a note to be given also by catechol. Procter ("Lab. Book," p. 152) has shown that this is an error, though a blue violet is given by resorcinol, which is often present in commercial catechol; and this correction is quoted by the author himself on p. 54 without remark!

Schiff's reputed syntheses of digallic acid, both with phosphorous oxychloride and with arsenic acid, are now generally discredited, as it has been found impossible to free the product from the reagents without reconverting it into gallic acid. Hunt's attempted synthesis with monobromo-proto-catechuic acid and potassium gallate (which is mentioned on p. 17 without the name of its author) has never been successfully repeated, though tried by Schiff, and is now assumed to have been an error. The formula for ellagic acid, which on pp. 19 and 23 is attributed to Nierenstein, was proposed long ago by Graebe, and proved by Kostanecky, Perkin, Herzig, and others. On pp. 29 and 30 all the information as to the chemistry of catechins is more or less obsolete; the work of A. G. Perkin is not quoted, and the constitution according to Kostanecky is not given, though this is one of the few formulæ in the chemistry of the tannins which deserves credit.

On p. 31 chestnut wood extract is said to contain 14 to 20 per cent. of tannin, though 30 per cent. is nearer the modern standard, and this criticism applies to nearly all the tabular information given on the strength of tanning materials, the analyses quoted being mostly by obsolete methods giving far too low results. In fact, the information on tanning materials, occupying pp. 31 to 55, is very sketchy and incomplete; for instance, oak-wood extract, which is now one of the most important materials, is mentioned only in a table of 1883 attributed to Simand, which gives the total tannin as 14·47, and the part of this soluble in cold water as 15·09! On pp. 42 and 43 qualitative tables attributed to Procter are really due to

Andreasch, and no mention is made of the fact that they are only applicable to alcoholic solutions, though the original source is referred to on p. 55 with apparently no idea that the tables are identical with those given. No mention is made of the frequent use of extract of mangrove bark as adulterant of quebracho, nor of methods for its detection.

The modern quantitative methods are correctly given, mostly in the words of the official prescriptions of the I.A.L.T.C. and the A.L.C.A., but are not critically discussed; and a good deal of space is occupied by the description of antiquated processes, most of which are known to give incorrect or unreliable results.

In the section on the analysis of leather the von Schroeder method of glucose estimation is very briefly described, but the necessary glucose table is omitted both there and in the section on glucose estimation in tanning materials, and in place of it a single factor is given. No mention is made of the ordinary volumetric method, which, if carried out with proper precautions, gives results of considerable accuracy, and is very useful as a rapid process of preliminary investigation, and will usually decide whether or not artificial loading has taken place. Sucrose is now sometimes used in place of glucose, and, of course, is not estimated by either process without previous inversion. No mention is made of Fahrion's method of determining a tannage value by boiling with water, and in the formula for calculating hide-substance from the Kjeldahl determination of nitrogen, + is written instead of ×. Further remarks seem unnecessary. HENRY R. PROCTER.

DYES AND COLOURING MATTERS, DYESTUFFS OF GROUPS 6 TO 12, COLOURING MATTERS OF NATURAL ORIGIN AND ANALYSIS OF COLOURING MATERIALS. By J. T. HEWITT.

A very valuable feature of this fourth edition of Allen's well-known treatise is that the analytical examination of the various organic products under consideration is preceded by thoroughly up-to-date descriptions of the systematic chemistry of these materials, each section being dealt with by competent experts. In the chapter on dyes and colouring matters a brief résumé is given of the modern development of artificial colouring matters and of the relations of dyes to the textile fibres. A consideration of the nitro-, nitroso-, and *iso*nitroso-colouring matters is included in this section, which also deals very fully with the important azo-dyes. Very comprehensive tables are given, showing the mode of formation and the specific reactions of the azo-dyes. In some instances, however, the abbreviations employed have almost ceased to be intelligible. For example, Fast Acid Scarlet $(4)SO_3Na.C_{10}H_6.N_2(6).C_{10}H_6.OH$ is said to be obtained from beta-acid, and Acid Ponceau from α and γ naphthylamines (*sic*). There are also a few orthographical errors, such as "unsoluble," "phenatidine," and "dinitrosoresorcnol."

The remaining synthetic dyes are arranged in seven groups (groups 6 to 12 inclusive), a separate section being devoted to indigo.

The prevailing views on the constitution of the di- and tri-phenylmethane colouring matters are critically examined and the properties of the principal commercial dyes of these series are arranged in tabular form. The azine, oxazine, quinoline, and acridine dyes are similarly treated.

Indigo is classified under colouring matters of natural origin, and, possibly on this account, very little space is devoted to the important subject of synthetic indigo. The estimation of indigotin is, however, dealt with in greater detail, especially as regards the oxidation and reduction tests, reference being made to such modern developments as the production of indigotintetrasulphonic acid and the reduction of sulphonated indigotin with titanous chloride. A survey of such natural dyestuffs as logwood, fustic, turmeric, cochineal and the red woods is followed by a chapter on the analysis of colouring materials, giving methods for the examination of commercial colouring matters and their detection of the dyed fibres. The detailed information contained in a very concise form in this section of the treatise should prove of the greatest service to analysts called upon to examine fabrics dyed with the colouring matters employed in modern practice. G. T. MORGAN.

FOODS: THEIR ORIGIN, COMPOSITION, AND MANUFACTURE. By WILLIAM TIBBLES, M.D., LL.D., etc. London: Baillière, Tindall and Cox. 1912. Price 18s. net.

This work is a veritable encylopædia of foods, and of substances used in the preparation of food. It would, indeed, be difficult to mention any food material, from the most important and necessary to the most trivial condiment or colouring matter, which does not here find a place and adequate description.

The hard-worked scientific man must always feel a sense of gratitude towards his more leisured confrère, when that leisure is employed in the collection and compilation of the widespread information which to-day forms so important an adjunct to the analytical armoury.

In order that such a work should fulfil a really helpful and useful purpose, it is necessary that the author should possess sufficient scientific insight to enable him to judge of the value of the different sources of information at his disposal and sufficient discrimination to sift them adequately. When these dicta are applied to the work under review, one is compelled to admit that they are fulfilled.

The selection of authorities is excellent, and the "drawing from opposing authorities the pith of their knowledge," described in the preface by the author as "a source of delight," has, in nearly all cases, been happily carried out.

It is almost unnecessary to say that no man can be an authority on all branches of the widely diverse subjects here presented, nor does it seem that the author makes any special claim to have any special knowledge of any one of them. It is, therefore, inevitable that the expert in any of these subjects may have cause to find fault with some of the statements, etc., made, particularly if his pet theories happen to be omitted, or inadequately mentioned. In actual fact there are numerous errors, but these do not militate against the intrinsic value of the work. The author is happiest in those sections which deal with processes which are not closely interwoven with trade secrets and methods, for which one will look in vain in this, as in any other book; but in the general composition of the final products, and in trade descriptions of these products, there is much sure ground.

One notes with pleasure the great use which has been made of the invaluable bulletins of the U.S. Department of Agriculture, and the large collection of these will be useful to many. It would be quite impossible to deal with the various chapters one by one, as the before-mentioned diversity of subject-matter precludes personal criticism as much as it precludes comprehensive expert treatment in its composition.

Part I. deals with the "chemical constitution of foods and the classification and character of their proximate principles." The matter dealing with this intricate subject is well put together, and displays knowledge of the latest work, though some references to the conclusions of older investigators might well have been omitted in view of more recent findings. The chemical presentation is somewhat crude in expression in many cases and this is true of the work as a whole, though there is never any real doubt as to the meaning intended. There are some curious and oft repeated errors which are difficult to understand, such as the description of "cholesterin" as a nitrogenous fat, though in its proper place it is correctly described. "Protagon" is also spoken of as a separate entity. The carbohydrates are fully dealt with, and the section has been compiled from reliable sources.

In the chapters on the Mammalia, Pisces, and Aves, the author is probably at his best, as the scientific descriptions and classification are from excellent authorities, and are presented in a concise and thorough manner, with full details as to distribution on the earth's surface, and to the use made of them as food. There is also a useful chapter on meat inspection and regulations, and on the composition of meat as eaten and its digestibility. The properties of meat extracts, soups, etc., are given, and from the remarks appended there is evidently considerable personal knowledge of these aliments.

The care which the author has expended in the sifting of available data is nowhere better seen than in the sections dealing with such debatable matters as milk and milk products. In addition to the general consideration of the subject, he has succeeded in disentangling from the masses of conflicting evidence which have gathered round such points as the influences which affect the composition of milk, the effect of feeding, period of lactation, etc., the more vital and probably correct conclusions. One notices, however, with a slight feeling of amusement, the list of constituents of milk which is given, and which, if all had to be determined, would render the analysis a rather titanic task, very different from the usual "total solids and fat" beyond which few analysts appear to venture.

The bacteriology of milk-souring is, on the other hand, very inadequately described, and the latest work is not mentioned.

Under milk preparations, cheese, both in its aspects of manufacture and subsequent ripening, is adequately dealt with, and nearly every milk preparation known finds notice, and, in general, correctly, except in the case of condensed milk, for which only one or two quite out-of-date methods are referred to.

The chapters on Cereals, Vegetables, and Fruits are excellent and most complete, the botanical descriptions being quite reliable. Full details are given of the preservation of fruit, and it is to be wished that the description of "jam" manufacture was applicable to more of our jams than it can possibly be. The Public Analyst will be fully in accord with the author in his remarks under "What is jam?" A comprehensive list of colouring matters is appended, and it is noticeable that some common errors in regard to these are avoided.

Even in such a difficult subject as that of oils, the information is reliable and trustworthy, though some of the processes stated to be used are somewhat obsolete. A few of the trade descriptions in this section are not quite correct, such as the information that "nucoline" is palm-kernel oil.

The other chapters which deal with tea, coffee, chocolate, spices, condiments, sugar, honey, confectionery, wines, spirits, and malt liquors may be passed over without comment.

The only serious mistake that the author has made is the inclusion of analytical methods, as in nearly all cases these are hopelessly impossible or badly described. As they are quite out of place, it would have been much better if they had been omitted. In spite of the condensed nature of the book, the matter is always in most readable form, and the proof-reading, with a few exceptions, carefully done.

There is not the least doubt that this work would be of much assistance to the general analyst as a concise book of reference. C. REVIS.

VOLUMETRIC ANALYSIS FOR STUDENTS OF PHARMACEUTICAL AND GENERAL CHEMISTRY. By C. H. HAMPSHIRE, B.Sc., A.I.C. Pp. vi + 104. London: J. and A. Churchill. 1912. Price 3s. 6d. net.

As its title indicates, this book is not intended for the use of analysts, but for the instruction of students, particularly pharmaceutical students, and it must be judged accordingly. For example, anyone who attempted to use this book as a book of reference, and followed the directions given on p. 98 for the analysis of a mixture of boric acid and borax, would soon find himself in difficulties, unless he already knew all that this book has to teach. But the student who has steadily worked through the book up to this point would be in no difficulty, for in earlier pages the principles underlying mixed titration are clearly explained. The whole book is a model of lucidity, and one can only regret that the author did not suppress some redundant paragraphs, such as the titration of sodium bicarbonate after that of potassium carbonate had been described, and so have found room for a short description of methods of standardising apparatus. Criticism on many points which at first sight seem to invite criticism, is silenced by the reflection that the author's students are mainly concerned to determine whether certain drugs comply with the requirements of a Pharmacopœia which, in addition to other demerits, is fourteen years old. There are very few misprints, but presumably the direction on p. 23 to determine the strength of lime-water by titrating 25 c.c. with "normal" acid must be reckoned as one, and one would like to believe that the word "better" on p. 6, line 4, was a misprint for "necessary." The former is scarcely a strong enough word with which to back a recommendation to make use of a desiccator for cooling ignited sodium carbonate, which is to be the ultimate standard in all the acidimetric and alkalimetric work which is to follow. G. CECIL JONES.

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SEPTEMBER, 1912 Vol. XXXVII., No. 438.

THE ANALYST.

OBITUARY.

BENJAMIN EDWARD REINA NEWLANDS.

Benjamin Edward Reina Newlands, youngest son of the Rev. William Newlands, was born in Southwark on January 23, 1842. On his mother's side he was of Italian descent. Both he and his brother John received their early education at home with their father, and subsequently at St. Saviour's Grammar School. The brothers studied chemistry under Professor A. W. Hofmann, and at an early age started an analytical practice in Great St. Helen's, London. Benjamin became assistant to Professor C. W. Heaton at Charing Cross Hospital, in 1862, and to Professor Way, of the Royal Agricultural Society, in 1863. His bent was always, however, towards technology, and at the end of 1863 he was appointed chemist to Messrs. James Gibbs and Co., manure makers, Victoria Docks, and from 1869 to 1873 he was manager at these works. In the latter year, he was made manager of Mr. James Duncan's Sugar Refinery, Clyde Wharf, Victoria Docks. This refinery was contiguous to Messrs. Gibbs's works, and while at the latter Newlands had had the *entrée*, and from time to time had given Mr. Duncan advice. He remained as manager, with his brother John as chemist, until the refinery, then the largest in the United Kingdom, closed in 1887, when the brothers once more embarked upon a professional career as analytical and consulting chemists at 27, Mincing Lane, London, and subsequently at 2, St. Dunstan's Hill, London. Well known as they were in the sugar trade, the brothers Newlands soon built up a good practice, mostly connected with sugar technology. Their consulting work was not confined to this country, their services being retained in India and the Colonies. A new edition of their treatise on "Sugar" has just been published. After the death of John Newlands in 1898, the writer was in partnership with Benjamin for five years. Benjamin Newlands was a Fellow of the Institute of Chemistry of Great Britain and Ireland, and had served on its Council and as a Vice-President. He was also a Fellow of the Chemical Society, and a member of our own Society. Of all scientific societies, he cared most for the Society of Chemical Industry, of which he was an original member, and served from the first on its Publication Committee, and, with a few intervals, on its Council. He was several times elected a Vice-President of the Society of Chemical Industry, and was Chairman of the London Section from 1895-96. He will be remembered by his professional brethren, not only by his wide practical knowledge, but no less by his sound common-sense, which was tempered by a characteristic geniality of manner. His fund of humour was wellnigh inexhaustible, and earned for him the reputation of the best *raconteur* in chemical circles. He died at his residence at Hayling Island on August 7.

Arthur R. Ling.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE ESTIMATION OF CARBON IN STEEL.

By A. G. LEVY, B.Sc., F.I.C.

THE direct combustion method for determining carbon in steel is now used so largely —many analysts using this method to the exclusion of all others, except for checking purposes—that it may not be amiss to point out one source of error hitherto apparently overlooked.

Whenever the direct process has been compared with the solution methods, it has been noticed that, except in cases where combustion appears to have been not quite complete, there is a tendency to slightly high results. Thus, many of the values quoted by Blount and Levy (ANALYST, 1909, **34**, 94) show a small excess over those obtained by the solution method. In the discussion on this paper, L. Archbutt (*ibid.*, p. 97) mentioned this tendency; and, more recently, Helen Isham (*J. Ind. and Eng. Chem.*, 1911, 3, 577) states that the values given by the direct method are usually 0·007 per cent. higher than those obtained by the use of the solution method. She also shows that there is no loss of carbonaceous gases during the dissolution of the steel in acidified potassium cupric chloride solution at *ordinary temperatures*.

It is obvious that if the sample of steel contains *dissolved carbonaceous gases*, such as carbon monoxide and dioxide, their carbon content will be included in its entirety in the values given by the direct combustion method; whilst it seems almost equally certain that it will not affect the solution process, since, even if the gases liberated are not expelled from the solution, they will pass with this into the filtrate from the insoluble carbonaceous residue, and leave the latter free from all but a possible trace of the double compound of cuprous chloride and carbon monoxide.

At first sight one might think that the quantities of gases involved are too small to affect the carbon determination. During the last few years, however, a good deal of work has been done on the amount and composition of the gases dissolved in steel, and it appears that errors of up to 0·02 per cent., or even more, may easily be due to this source. For the present purpose, by far the most valuable is a paper by E. Goutal (*Rev. Métallurgie*, Mémoires, 1910, vol. vii., p. 8). Goutal's method consisted in dissolving the sample in carefully boiled out acid potassium cupric chloride solution in a current of nitrogen, finally raising the solution to the boiling-point, and determining carbon monoxide, carbon dioxide, and sometimes other gases, in the gases evolved, and carbon in the residue. Proceeding in this way, he obtained the following results:

	CO_2. Per Cent.	CO. Per Cent.	C in Residue. Per Cent.	C (corresponding to Carbonaceous Gases). Per Cent.
Soft steel	0·0254	0·0063	0·160	0·0096
Medium steel	0·0210	0·0120	0·295	0·0108
Hard steel	0·0678	0·0137	0·596	0·0244
Extra hard steel	0·0792	0·0142	1·334	0·0277
Nickel steel (Ni, 2·04 per cent.)	0·0530	0·0060	0·340	0·0170
Chrome steel (Cr, 5·10 per cent.)	0·0510	0·0115	0·360	0·0188
Basic open hearth iron ...	0·1890	0·0132	4·110	0·0571

The values in the last column are not given by Goutal.

He also shows that, if the solution contains not more than 10 per cent. of HCl, and the temperature is not raised above 45° C., the CO_2 remains wholly in the liquid, whilst the CO is partly disengaged. Thus, working with 10 grms. of a steel containing 0·95 per cent. carbon, he found:

	CO_2. Per Cent.	CO. Per Cent.	C (corresponding to Carbonaceous Gases). Per Cent.
During dissolution	0·0000	0·0068	0·0029
In filtrate (on boiling) ...	0·0075	0·0070	0·0050
In residue (boiled with very dilute HCl)	0·0751	0·0015	0·0211
Total	0·0826	0·0153	0·0290
Check by first method ...	0·0829	0·0153	—

The carbon dioxide here found to remain with the residue would probably be largely, if not wholly, expelled during the drying usual before burning the residue, and hence would be lost in the solution method.

In another series of experiments he obtained the following values:

	Medium Steel. Per Cent.	Hard Steel. Per Cent.	Extra Hard Steel. Per Cent.
C in residue	0·2950	0·5960	1·3340
C in CO_2	0·0057	0·0185	0·0216
C in CO	0·0051	0·0059	0·0061
C in cases not identified ...	0·0009	0·0171	0·0158
C total	0·3067	0·6375	1·3775

As far as my reading goes, Goutal is the only author who mentions the influence of the dissolved gases on the two methods of estimating carbon. Thus, on p. 13 (*op. cit.*) he states quite incidentally that he prefers his own method of determining carbon (to the direct combustion method), because it enables one to distinguish between two forms of carbon (that in the residue and that evolved as gas), which influence the properties of the steel in quite different ways. This general silence is all the more striking when one reflects that the same consideration applies to all the numerous *wet* methods of combustion.

Further proof of the existence of appreciable quantities of dissolved carbonaceous gases in steel and iron is found in several other papers by different authors, all of whom determined the gases evolved on heating the samples *in vacuo*. A number of recent determinations are collected in the table following, in which I have calculated in the last column the percentage of carbon corresponding to the carbonaceous gases evolved:

Reference.	Nature of Sample.	C.	Weight of Sample	Gases evolved on Heating *in vacuo*.							Corresponding to Carbonaceous Gases.
				Total.	CO_2.	CO.	H.	N.	CH_4.	Not determined.	
		%	Grms.	Gases in c.c. as measured at 0° and 760 mm.							%
1	Wire, 1 mm. diameter ...	0·074	40·000	36·43	7·010	20·33	7·83	1·36	—	—	0·037
1	Wire, 0·5 mm. diameter	0·047	34·000	33·70	6·220	12·70	11·89	2·89	—	—	0·030
1	Foil, 1 mm. thick ...	0·130	82·500	78·75	5·080	53·46	17·40	2·81	—	—	0·038
1	Filings	0·099	46·000	106·00	5·160	66·45	29·17	5·22	—	—	0·084
2	Planings, mixed... ...	0·120	—	25·43	3·690	13·49	7·31	0·76	—	0·18	—
2	Planings, surface ...	0·120	75·000	11·09	0·590	3·84	5·67	0·67	—	0·32	0·003
2	Planings, half-way ...	0·120	75·000	16·74	1·400	10·37	3·67	1·15	—	0·25	0·008
2	Planings, centre... ...	0·120	75·000	11·46	0·880	2·72	4·78	0·41	—	2·67	0·003
				Gases in per Cent. of Total Gas.							
				c.c.	%	%	%	%	%	%	
3	Planings, 45 per cent. nickel steel	0·150	50·000	21·15	7·000	65·80	20·92	6·28	—	0·16	0·078
		0·150	50·000	61·97	6·500	47·80	42·10	3·70	—	—	0·058
				Gases in c.c. as measured at 0° and 760 mm.							
4	Piece without blowholes	0·900	69·310	91·86	1·540	41·83	47·77	0·07	0·66	—	0·034
4	Piece with blowholes ...	0·810	63·261	42·09	0·370	17·85	22·99	0·20	0·73	—	0·016
				Gases in c.c. at 0° and 760 mm. per c.c. of Metal.							
5	Planings, basic Bessemer steel	0·050	—	—	0·150	2·36	0·20	0·55	—	—	0·017
		to	—	—	0·004	0·91	0·13	0·16	—	—	0·006
		0·080	—	—	0·220	1·63	0·30	0·35	—	—	0·013
5	Electric furnace steel ...	0·150	—	—	0·690	1·49	0·22	0·10	—	—	0·015
5	Crucible steel, 0·24 per cent. wide	0·400	—	—	0·020	2·45	0·18	0·35	—	—	0·017
		0·400	—	—	0·020	1·31	0·35	0·22	—	—	0·009
		0·400	—	—	0·010	1·07	0·11	0·31	—	—	0·007
5	Rail steel	—	—	—	0·220	4·45	0·51	0·92	—	—	0·031
5	Swedish pig-iron ...	3·650	—	—	0·950	5·80	0·91	0·56	—	—	0·046

REFERENCES TO TABLE.

1. O. Boudouard, *Rev. Métallurgie*, Mém., 1908, **5**, 73.
2. G. Belloc, *Rev. Métallurgie*, Mém., 1908, **5**, 479 and 482.
3. G. Belloc, *Rev. Métallurgie*, Mém., 1908, **5**, 573 and 574.
4. T. Baker, *J. Iron and Steel Inst.*, Carnegie Schol. Mem., 1909, **1**, 225.
5. P. Goerens, *Rev. Métallurgie*, 1910, **7**, 392.

With the exception of the two results due to Baker, all the determinations were carried out on planings or thin wire and foil. Hence, gas evolved from large blowholes is excluded, and the results apply directly to samples as usually taken for direct combustion. It will be seen that plus errors of 0·03 per cent. carbon, due to this source alone, are easily possible, even if four higher values (up to 0·08) are rejected as abnormal. Incidentally, the presence of small quantities of hydrogen in the metals supplies a reason for the insertion of a drying tube between combustion tube and potash bulbs, which precaution is occasionally omitted. Regarding the accuracy of the determinations quoted, it may be mentioned that Baker found a small furnace blank of about 3 c.c. total gas, consisting largely of hydrogen and nitrogen, due probably to the walls of the heated porcelain tubes not being perfectly impervious to gases at high temperatures under the high *vacua* used.

Finally, to sum up, when working to a close carbon specification, it may not be a matter of indifference to the maker of the steel whether the analyst uses a solution or a direct combustion method.

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THE BACTERIOLOGY OF AERATED WATER.

By G. D. ELSDON, B.Sc., A.I.C., and NORMAN EVERS, B.Sc., A.I.C.

During the past few years attention has been drawn to the bacteriological examination of commercial soda-water, and a number of results have been published by Public Analysts and others—*e.g.*, Klein, Report of the Medical Officer of Health for the City of London (Analyst, 1911, 36, 16). Thirty-seven samples of commercial soda-water have recently been completely analysed in this laboratory, with the following bacteriological results: In no case has typical *B. coli* (flagniac) been found in 50 c.c. In two cases fermentation was obtained in glucose taurocholate with 50 c.c., in two cases with 10 c.c., and in 1 case with 1 c.c.; but in all these cases further examination showed that these were not typical *B. coli*. The number of organisms growing on jelly in three days, found in twenty-five samples stored in siphons, varied from 1 to 150 per c.c.; whilst the number found in waters stored in bottles, all precautions being taken to prevent the access of bacteria during opening, was found to be in some cases 1,000 to 3,000 per c.c.

It seemed desirable to find out how far these bacteriological results, obtained in many cases probably several days (and possibly weeks) after bottling, are of value as an indication of the purity or otherwise of the original water, for it seemed probable that the presence of carbon dioxide under pressure would have a deleterious action on the micro-organisms present in the water. For the purpose of carrying out these experiments, the apparatus known as the "Prana Sparklet" siphon was used. The siphons were thoroughly cleaned and steamed for one hour, with careful heating and cooling to prevent breakages, on each of two successive days, the siphon-head being plugged with wool. The water to be experimented upon was then run in by means of a sterile funnel, and carbonated in the usual manner. Small quantities were withdrawn from the siphon from time to time for the purpose of bacteriological examination, estimations being also carried out on the water itself as a control.

Such experiments were carried out on—(1) the number of organisms growing on jelly at 21° C., counting after three days; (2) the number of organisms growing on neutral-red lactose taurocholate agar (Rebipel agar) at 37° C., counting after two days; and (3) the power of fermenting glucose taurocholate broth.

1. *The Number of Organisms growing on Jelly at 21° C.*—Various samples of water were taken and aerated as described above, and the number of organisms per c.c. growing on jelly at 21° C. estimated after various lengths of time in both the carbonated and uncarbonated water. The results obtained are given in the following table:

TABLE I.

Source of Water.	Number of Bacteria growing on Jelly in Three Days at 21° C.				
	Original Water.	After Three Days.		After Seven Days.	
		Control.	Carbonated.	Control.	Carbonated.
Filtered river	950	910	125	867	93
Filtered upland surface... ...	19	845	15	4,200	1
Unfiltered upland surface ...	391	Liq.	3	5,500	1
,, ,, ,, ...	3,400	7,300	53	7,400	44
,, ,, ,, ...	290	2,650	63	1,700	51
,, ,, ,, ...	10,000	36,800	397	33,900	142
Deep well	Liq.	240,000	950	—	—

These results, obtained from different types of water, show that the total number of organisms is rapidly reduced by carbonation, and in well-filtered waters, where the number of organisms is small originally, carbonation prevents these organisms from multiplying. In the case of the polluted water, it will be observed that the number of organisms was very rapidly reduced.

2. *Number of Bacteria growing on Rebipel Agar at 37° C. in Two Days.*—Sterile tap-water was in two cases inoculated with a small number of *B. coli* (flaginac) grown in peptone water for two days. Part of the water was aerated, and the effect of aeration observed as before.

TABLE II.

Original Water.	After Three Days.		After Seven Days.		After Fourteen Days.	
	Control.	Carbonated.	Control.	Carbonated.	Control.	Carbonated.
63	62	58	57	25	46	5
7,000	—	—	6,200	38	6,100	5

Experiments were then undertaken on the number of organisms growing on Rebipel agar which are naturally present in waters. The method used was similar to the one used under Section I. The results were as follows:

TABLE III.

Source of Water.	Number of Bacteria growing on Rebipel Agar at 37° C.				
	Original Water.	After Three Days.		After Seven Days.	
		Control.	Carbonated.	Control.	Carbonated.
Deep well water	275	1,820	66	—	—
" " "	108	44	19	8	7
River water	347	714	23	905	2

3. *Effect of the Addition of Sodium Bicarbonate on the Fermentation of Taurocholate.*—Fifty c.c. of sterile carbonated water were added to taurocholate, and then inoculated with a little cultivated *B. coli* (flaginac) and incubated at 37° C. After twenty-four hours a vigorous acid and gas production was observed.

Fifty c.c. of sterile carbonated water containing 10 grains per pint of sodium bicarbonate were added to taurocholate and inoculated as above. After twenty-four hours a vigorous growth was observed.

Fifty c.c. of a well-water known to contain *B. coli* were taken, and sufficient sodium bicarbonate added to make the alkalinity equal to 10 grains per pint. The water was then added to taurocholate, and after incubating at 37° C. for twenty-four hours, a similar growth was obtained to that given by the original water.

Fifty c.c. of carbonated water containing sodium bicarbonate equal to about 5 grains per pint were put into taurocholate; after twenty-four hours a vigorous growth was observed.

These results show that the growth of bacteria in glucose taurocholate is prevented neither by an alkalinity of 10 grains per pint of sodium bicarbonate, nor by carbonation, nor by both together.

4. *Organisms producing Acid and Gas in Glucose Taurocholate.*—The results given below were obtained by growing natural waters in glucose taurocholate with and without carbonation. In the table, (*a*) signifies the carbonated water, whilst (*b*) signifies the uncarbonated or control water.

TABLE IV.

Well Water A.

		50 c.c.	10 c.c.	1·0 c.c.	0·1 c.c.
Original		+	+	+	−
After four days	(*a*)	+	+	−	−
	(*b*)	+	+	−	−
After seven days	(*a*)	+	−		
	(*b*)	+	−		

Well Water B.

		50 c.c.	10 c.c.	1·0 c.c.	0·1 c.c.
Original		+	+	+	
After four days	(a)		+	+	
	(b)		+	+	
After seven days ...	(a)	+	+	+	
	(b)	+	+	+	
After twelve days	(a)				
	(b)		+		

Well Water C.

		50 c.c.	10 c.c	1·0 c.c.	0·1 c.c.
Original		+	+	+	+
After four days	(a)		+	+	+
	(b)		+	+	+
After seven days	(a)		+	+	+
	(b)		+	+	+
After twelve days	(a)		+	+	+
	(b)		+	+	+

It will be seen that except in the case of the well water B the carbonation did not sensibly affect the organisms fermenting glucose taurocholate in the time given.

Conclusions.—The general conclusions to be drawn from these experiments seem to be the following:

(*a*) The total number of organisms growing on jelly at 21° C. is considerably reduced by carbonation, and a water containing originally a large number of organisms may, after a week's standing under pressure, contain only a number such as would be found in a very good water.

(*b*) *B. coli* are not so rapidly reduced as other organisms, but die off rather more rapidly than in ordinary water.

(*c*) The number of organisms growing on Rebipel agar are much reduced by carbonation, especially those which do not produce the typical *B. coli* red appearance. This latter fact was shown by the appearance of the plates after incubation.

(*d*) Ten grains per pint of sodium bicarbonate does not affect the growth of organisms in glucose taurocholate; neither does excess of carbon dioxide, such as is found in carbonated waters.

It follows, therefore, that bacteriological results obtained on a water which has been carbonated for more than twenty-four hours are of little value as an indication of the source or purity of the original water, except in the case of growth in glucose taurocholate. In this latter case correct results may probably be obtained for a week after carbonation.

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SOLUBILITIES OF THE LEAD SALTS OF THE HIGHER FATTY ACIDS IN ETHER AND IN PETROLEUM ETHER.*

By G. B. NEAVE, M.A., D.Sc.

Owing to the fact that the solubility of lead oleate in ether is employed in estimating the amount of oleic acid in a mixture, and the possibility that errors may arise from the solution of the lead salts of saturated acids, the latter were prepared and their solubilities in ether and petroleum ether determined.

Preparation of the Lead Salts.—The pure acid was neutralised with ammonia, and the lead salt precipitated by addition of a saturated solution of lead acetate. The lead salt was filtered, washed, and recrystallised from alcohol.

Lead caproate	m.pt.	73-74° C.	Pb found	47·23 %	Pb calculated	47·37 %
Lead heptylate	„	90·5-91·5° C.	„	44·27 %	„	44·51 %
Lead caprylate	„	83·5-84·5° C.		—		—
Lead nonylate	„	94-95° C.	„	39·68 %	„	39·73 %
Lead caprate	„	100° C.	„	37·59 %	„	37·70 %
Lead laurate	„	103-104° C.	„	34·16 %	„	34·21 %
Lead myristate	„	107° C.	„	31·34 %	„	31·32 %
Lead palmitate	„	112° C.		—		—
Lead stearate	„	125° C.		—		—

Lead Oleate.—This salt was obtained as a white powder, m.pt. 45-50° C., which is considerably lower than that quoted by Gottlieb (*Annalen*, 1845, 57, 38). Other preparations were made, but all the specimens gave approximately the same m.pt. The lead oleate thus obtained was extremely soluble in ether and in petroleum ether.

Attempts were also made to prepare the lead salts of other unsaturated acids. These, however, were very rapidly oxidised on exposure to the air.

Determination of Solubilities of the Lead Salts.—The ether employed was purified by distillation over sodium; the petroleum ether was the fraction collected from 40-60° C. The flask containing the lead salt and the solvent was placed in a thermostat maintained at a temperature of 20° C., and was fitted with a stirrer driven by a small motor. Since filtration by the ordinary method was slow and involved a loss of solvent, a special pressure-filtering apparatus was used. The flask containing the solution and excess of lead salt was closed with a cork, through which passed, in addition to the stirrer, two tubes, one terminating just below the cork, the other at the bottom. The second tube, communicating with a weighed flask, was widened at the end, on which was the filter, consisting of two layers of Swedish filter-paper, with an outer layer of thin silk. After the solution was saturated, dry air was forced into the flask, and any convenient volume collected in the weighed flask and evaporated to complete dryness. The solubilities at the boiling-points of the solvents were determined in the same manner, the first flask being attached to an efficient condenser.

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* This work was carried out under the conditions of the Analytical Investigation Scheme.

The solubilities in the following table are expressed in grms. per 100 c.c. solution:

	Ether.		Petroleum Ether.	
	20° C.	Boiling-Point.	20° C.	Boiling-Point.
Lead caproate ...	—	1·3640	—	0·0608
Lead heptylate...	0·2397	1·4900	0·0200	0·0528
Lead caprylate...	0·0938	0·5460	practically insoluble	0·0384
Lead nonylate ...	0·1115	0·2404	practically insoluble	0·0450
Lead caprate ...	0·0290	0·4285	practically insoluble	0·0170
Lead myristate...	practically insoluble	0·0555	practically insoluble	0·0210
Lead laurate ...	practically insoluble	0·0205	practically insoluble	practically insoluble
Lead palmitate...	practically insoluble	0·0261	practically insoluble	practically insoluble
Lead stearate ...	practically insoluble	practically insoluble	practically insoluble	0·0170

It will be seen from the table that the solubilities are very small, while that of lead oleate, as stated previously, is very great.

I desire to thank Messrs. E. Anderson. A.I.C., A. P. Clark, D. Hamilton, A.I.C., and J. Porter, A.I.C., for their assistance in this work.

THE ROYAL TECHNICAL COLLEGE,
GLASGOW.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

ANALYSIS OF FOOD AND DRUGS.

Micro-Chemical Tests for the Identification of Some of the Alkaloids. E. B. Putt. (*J. Ind. and Eng. Chem.*, 1912, 4, 508-512.)—In the method described, a fragment of the alkaloid was dissolved on a microscope slide in a drop of dilute hydrochloric acid, a drop of the reagent was added, and the nature of the precipitate examined under the microscope. In many cases distinct types of crystals were formed successively by the same precipitate. Some forty different alkaloids and other substances were thus tested, and a description is given of the crystalline or amorphous precipitates obtained. The paper is illustrated with photo-micrographs.

W. P. S.

New Method of Estimating Caffeine. G. Costes. (*Ann. Chim. anal.*, 1912, **17**, 246-249.)—The method is based upon the fact that caffeine is not destroyed by three hours' heating with sulphuric acid, and after the destruction of the bulk of the accompanying organic matter, the alkaloid may be extracted from the neutralised solution with chloroform. Twenty grms. of the ground coffee (or more in the case of samples containing only about 0·1 per cent. of caffeine) are mixed little by little with 15 to 20 c.c. of pure sulphuric acid (sp. gr. 1·835), and the flask placed for ten to fifteen minutes on the water-bath at 100° C., after which the mixture is extracted with three successive portions (about 200 c.c., 150 c.c., and 100 c.c.) of boiling water, the extraction being continued about ten minutes the first time and five minutes the second and third time. After each extraction the liquid is filtered into a porcelain basin containing a little sodium hydroxide (insufficient to neutralise the whole of the acid). The united filtrates are rendered slightly alkaline with sodium carbonate, concentrated to about 200 c.c., filtered if necessary, and extracted three times with chloroform (50 c.c., 35 c.c., and 30 c.c.). The chloroform extracts are united and distilled, and the residue of caffeine purified by heating it for ten minutes with 2 c.c. of sulphuric acid on the water-bath, and adding just sufficient water to render filtration possible. The filter is washed five or six times with a little boiling water, and the filtrate and washings rendered slightly alkaline with ammonia, cooled, and extracted with three successive portions of chloroform (25 c.c., 20 c.c., and 15 c.c.). The extracts are evaporated in a tared basin on the water-bath at about 80° C., and the residue is dried for thirty minutes at 100° C. and weighed. However white the crystals appear, the residue will not contain more than about 93 per cent. of pure caffeine, and it is therefore advisable to estimate the nitrogen by Kjeldahl's method, and to calculate the corresponding amount of caffeine. For industrial purposes results in close agreement with those given by the methods of Granval and Lajoux and of Helger and Juckenack may be obtained by omitting the neutralisation of the liquid after the initial treatment with sulphuric acid and filtration, extracting the caffeine with (1) 100 c.c., (2) 100 c.c., and (3) 75 c.c. of chloroform, evaporating the extracts, and weighing the residue.

C. A. M.

Comparative Value of the Catalase, Reductase, and Leucocyte Tests in Detecting Mastitis. O. Gratz and A. Náray. (*Milchwirtschaftl. Zentralb.*, 1912, **41**, 225-232, 257-263, 289-303; through *Chem. Zentralb.*, 1912, II., 287.)—The milk of 153 individual cows was examined, re-examined after ten or twelve days, and the examination repeated several times in all doubtful cases. As a result the reductase test, whether in Barthel's original form or Schardinger's modification of it, is declared to be too uncertain to be of value. The catalase test is said to be five to ten times as sensitive as the leucocyte test, but the latter is necessary to control the results of the former in cases where the abnormal catalase number is due to blood. In such cases the blood will usually be detected by the reddish colour of the sediment in the capillary of the tube used for collecting the sediment for a leucocyte count. For the catalase test the authors use Koestler's apparatus, 10 c.c. of milk, and 5 c.c. of 1 per cent. hydrogen peroxide solution, and allow two hours at [illegible] to

24° C. They find that the test readily fixes suspicion on the mixed milk of ten to twenty cows, only one of which is suffering from mastitis. G. C. J.

Estimation of Husk in Cocoa Powders. L. Kalusky. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **23**, 654-661.)—The method proposed depends on the difference between the sp. gr. of the cell tissue of the inner portion of the cocoa bean and that of the husk. The sp. gr. of the cell tissue of the inner portion (obtained by treating the fat-free substance with diastase, and subsequently with hydrochloric acid) varies from 1·1131 to 1·3503, and is usually less than 1·25; whilst that of the husk lies between 1·4324 and 1·9337, and in the majority of cases is upwards of 1·50. The authoress found that it was possible to separate the two tissues by means of a mixture consisting of chloral hydrate, glycerol, and water, and having a sp. gr. of 1·415 at 17° C., and to estimate within narrow limits the quantity of husk which had been added to cocoa powders, when both the powder and the husk had been prepared by herself from cocoa beans. When commercial cocoa powders were examined it was found, however, that the sp. gr. of the cell tissue of the inner portion was somewhat higher than the above-mentioned figures, and it was found necessary to employ a medium having a sp. gr. of 1·5 in order to separate the two kinds of tissue. The details of the process as applied to the estimation of added husk in cocoa powder are as follows: About 5 grms. of the cocoa are extracted with ether until the fat has been removed completely. One grm. of the fat-free, dry powder is boiled for five minutes with 500 c.c. of water, then cooled to 65° C., and maintained at this temperature for one hour after the addition of a small quantity of diastase. Twenty-five c.c. of 25 per cent. hydrochloric acid are next added; the mixture is boiled for thirty minutes, when the insoluble tissue is collected on a weighed filter, washed with hot water, dried for two hours at 100° C., and weighed. The dry tissue is detached from the filter, ground as finely as possible in a mortar, again dried, and a weighed portion of it mixed in a suitable tube with 20 c.c. of the medium, and submitted to centrifugal action for five minutes. The medium is prepared by mixing 210 grms. of chloral hydrate, 50 grms. of glycerol, and 35 grms. of water, and should have a sp. gr. of 1·50 at 17° C.; if this is not the case, more chloral hydrate or water is added, as required. The portion of tissue which is at the bottom of the tube is now separated, mixed with 100 c.c. of water, collected on a filter, washed with hot water, dried, and weighed. A convenient form of tube for use in the centrifugal operation is described; it consists of a glass tube which is drawn out to a narrow point at the lower end, a small opening in this end being closed by a glass rod passing down the centre of the tube. The end of the rod is ground to fit the opening, and the sediment may be discharged from the tube by raising the rod slightly. The weight of heavy tissue thus obtained is calculated as a percentage quantity of the original cocoa, and the result is multiplied by 3 in order to give the percentage of husk present. This factor is obtained as the result of estimations of heavy tissue in samples of cocoa husk, the latter yielding on an average 30 per cent. of the tissue. When husk is absent, the whole of the tissue is found floating on the surface of the medium after the mixture has been submitted to centrifugal action. W. P. S.

Detection of Formic Acid in Fruit Products. F. L. Shannon. (*J. Ind. and Eng. Chem.*, 1912, 4, 526-528.)—The authors find that numerous substances, known to be free from formic acid, yield, when submitted to steam distillation, a distillate which reduces both silver nitrate and mercuric chloride. From none of these substances, however, could formic acid be isolated and identified as such. For the identification of formic acid the authors recommend that the acid be converted into formaldehyde by means of magnesium and dilute sulphuric acid; the acid may also be neutralised with lead hydroxide, when characteristic crystals of lead formate are obtained. In testing fruit juice or fruits, about 500 grms. of the sample are submitted to steam distillation, until the distillate ceases to give an acid reaction towards litmus (usually about 2,500 c.c. must be collected). The distillate is then neutralised, evaporated to 50 c.c., transferred to a flask provided with a long tube, to act as an air-condenser, and treated at the ordinary temperature with magnesium and sulphuric acid. The mixture is now distilled, and the first 10 c.c. of distillate tested for the presence of formaldehyde. Another portion of the sample may be distilled as before, and the distillate collected in a flask containing lead-cream (prepared by treating lead nitrate solution with potassium hydroxide until the mixture is faintly alkaline to phenolphthaleïn). The distillate is then evaporated to 50 c.c., filtered, and the filtrate set aside to crystallise. Any crystals obtained are washed with alcohol, to remove lead acetate, and then examined for formic acid by the usual tests (reducing reactions, conversion into formaldehyde, etc.). W. P. S.

Bromine Absorption of Certain Vegetable Oils and Fats. H. Sprinkmeyer and A. Diedrichs. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, 23, 679-687.)—The following percentage quantities of insoluble bromine compounds were obtained on subjecting various oils and fats to the process described by Hehner and Mitchell (ANALYST, 1898, 23, 315): Linseed oil, 28·9; candlenut oil, 8·8; hempseed oil, 8·82; walnut oil, 2·22; soja bean oil, 3·62; sesame oil, 0·14; mustard oil, 1·30; rape oil, 1·92. Poppy-seed oil, sunflower-seed oil, maize oil, cottonseed oil, castor oil, tea oil, earthnut oil, cocoanut oil, palm oil, palm-kernel oil, cacao butter, stillingia tallow, tulucuna fat, dika fat, and malukang butter, did not yield an insoluble bromine compound. In the case of shea butter, different specimens of the fat yielded from 5·2 to 8·6 per cent. of bromine compound, whilst mowrah butter gave 0·82 per cent., enkabang tallow 0·17 per cent., and adjab fat 2·2 per cent. As regards the four last-mentioned fats, the bromine compound appears to be formed from the unsaponifiable matters present, the fatty acids derived from the fats yielding no bromine compound after they have been freed from unsaponifiable matter. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Lactic Acid in the Presence of Proteins. J. Mondschein. (*Biochem. Zeitsch.*, 1912, 42, 105-123.)—Lactic acid is an important constituent of animal organs, but the greater part of this acid found in extracts of the organs is produced by post-mortem changes in the tissue, only a small quantity being a constituent of the living organ. Experiments carried out by the author show that when animal tissue is boiled with water only about two-thirds of the quantity of the lactic acid passes into the aqueous solution, the remaining third combining with the coagulated albumin. The following method is proposed for the estimation of the total amount of lactic acid present in an animal tissue or serum: In the case of muscular tissue, 50 grms. of the finely-divided substance are suspended in about 80 c.c. of water, the mixture is heated to boiling, and the coagulum is collected on a filter and washed with hot water until free from acidity. The filtrate is then titrated with $\frac{N}{10}$ alkali solution, using phenolphthaleïn as the indicator, and the acidity is expressed in terms of lactic acid. The coagulum is now removed from the filter, mixed in a beaker with 50 c.c. of water and 10 c.c. of a 10 per cent. sodium hydroxide solution, and the mixture is heated until fluid. One hundred c.c. of saturated sodium chloride solution are then added, the whole is again heated to boiling, and the hot mixture is saturated with sodium chloride. The precipitated proteins are collected on a filter and washed with hot saturated sodium chloride solution; the filtrate is rendered slightly acid with sulphuric acid, diluted to 500 c.c., filtered through a dry filter, and the lactic acid estimated in 250 c.c. of the filtrate by the method described by von Furth and Charnass (ANALYST, 1910, **35**, 445). The quantity of lactic acid thus found is added to that obtained by titration in the first part of the process, in order to give the total amount of the acid present in the substance under examination. Before applying von Furth and Charnass's method, it is advisable to boil the acid filtrate for ten minutes, in order to expel any hydrogen sulphide which may be present. The small quantity of protein which is precipitated on the addition of the sulphuric acid does not interfere with the estimation.

W. P. S.

Application of the Quantitative Precipitin Reaction in Honey Analysis —II. J. Thöni. (*Mitt. Lebensmitteluntersuch. u. Hyg.*, 1912, **3**, 74-94; through *Chem. Zentralbl.*, 1912, II., 151-153; *cf.* also ANALYST, 1911, **36**, 456.)—The author has studied the effect of heating the honey on the results of the precipitin reaction. Honey may be heated for various reasons—*e.g.*, for removing air-bubbles (temperature 35° C.), melting the honey out of the combs (temperature 50° C.)—and comparatively high temperatures are used in clarifying foreign crude honeys. The precipitin reaction depends on protein constituents which only begin to change at temperatures above 50° C., so that only such temperatures come into account. Experiments made at 60° to 98° C. showed that only temperatures a few degrees below 100° C. caused a diminution in the quantity of the precipitate, and then only if maintained for a certain length of time (ten to sixty minutes). Complete absence of the precipitate is only observed when the honey has been heated for longer than one hour at 100° C. At such a temperature the aroma and enzymes are destroyed, and such honey could not be regarded as normal. In the quantitative precipitin reaction the values obtained have only a relative significance, since they may vary according to the equivalent of the antiserum; only results obtained at the same time with the same serum are comparable among themselves. With a large number of samples of genuine honey of different origins no considerable differences in the variations of the precipitate values were established; these variations amounted to about ± 40 per cent. of the mean value. In the case of abnormal samples the serological method permitted an accurate identification of the honey. For the estimation of the catalase value of honey, 7·5 c.c. of honey solution (50 per cent.) were mixed with 7·5 c.c. of a 1 per cent. solution of hydrogen peroxide, and the volume of oxygen was observed after twenty-four hours. The author agrees with Witte that the catalase value alone is only of use in extreme cases as an indication of large adulteration, or incipient fermentation, or strong heating. As regards the effect of heating on the diastase value, the author considers that the original honey should be heated, not its solution, and in such cases a deficiency in diastatic activity only occurs at temperatures above 90° C., a higher limit than is found when the diluted honey is heated. For carrying out Fiehe's reaction, about 7 or 8 grms. of honey are dissolved in an equal quantity of water, extracted with ether; the clear, filtered ethereal solution is evaporated in a porcelain dish at 40° to 45° C., and the residue tested in the usual manner. Genuine honey does not give Fiehe's reaction; adulterated honey does; so that this is a reliable test for certain kinds of adulteration (*cf.* ANALYST, 1911, **36**, 456). J. F. B.

Determination of the Digestive Value of Papain. J. R. Rippetoe. (*J. Ind. and Eng. Chem.*, 1912, **4**, 517-518.)—Graber (ANALYST, 1912, 57) described a method for testing the digestive value of papain, using finely-ground steak suspended in 0·3 per cent. hydrochloric acid, and criticised the use of egg albumin in an alkaline medium. The author describes a process he has long employed for testing papain, in which 10 grms. of egg albumin is subjected to the action of 0·1 and 0·2 grm. of papain in 40 c.c. of 0·1 per cent. sodium hydroxide solution for six hours at 52° C. in a stoppered bottle, shaking every ten minutes. The whole is then washed into a graduated cylinder, made up to 70 c.c., and the volume of the deposit read at the end of one and sixteen hours. The following figures show the comparative effects of acid and alkali on the same sample of papain:

	Deposit in c.c. after standing.	
	One Hour.	Sixteen Hours.
Blank	52	43
0·1 grm. papain, 0·1 per cent. NaOH	26	18
0·2 ,, ,, 0·1 ,, ,, ...	8	8
0·2 ,, ,, 0·1 per cent. HCl	16	20
0·2 ,, ,, 0·2 ,, ,, ...	30	30
0·2 ,, ,, 0·3 ,, ,, ...	32	30

The inhibitory effect of the acid on proteolysis is clearly shown. H. F. E. H.

Laboratory Studies of Rennin. A. Zimmerman. (*J. Ind. and Eng. Chem.*, 1912, **4**, 506-508.)—Rennin precipitated with sodium chloride at 40° C. is sold in powder form mixed with lactose, and so prepared gives a much smaller yield than with sodium sulphate, but its activity is from five to six times greater. It has, when thus diluted, an activity of 1 : 30,000 or 40,000 within twelve minutes, contains more or less albumose, and is acid in reaction. Scale rennin is prepared by scaling the whole clarified rennet solution at a temperature of 43·5° C., and has a strength of 1 : 30,000 or 40,000. Scale rennin contains peptones and albumoses formed during the process of the digestion of the rennets with hydrochloric acid which it retains, having thus an acid reaction. The former product is the less permanent, this quality being apparently dependent on phosphoric acid, originally present in the rennets as the calcium salt, and set free by the hydrochloric acid when employed. The scale preparation, but not the sodium chloride product, gives the reaction of phosphoric acid, and the addition of this acid in the proportion of ·075 per cent. to new milk greatly accelerates the action of rennin. As milk gets older, rennin acts more readily. The curd in milk four to eight hours old forms very gradually, and when complete is not nearly so firm as that produced in the presence of phosphoric acid which materially reduces the time of action. Lactic acid has about one-fifth, and hydrochloric and oxalic acids one-tenth, the activity of phosphoric acid in this connection. Milk which is slightly acid curds more readily, and can be modified to require a longer time by addition of small quantities of alkali. To control the variability of milk for the assay of rennin, a standard rennin is required to show whether the milk is fast or slow. The action of the rennin is tested in not less than twelve different samples of milk. If the rennin is standardised to 1 : 30,000 in eight minutes, its use as a control with the rennin to be assayed shows whether the milk is fast or slow, and allowance is made accordingly. The rennin should be added to the milk as soon as the temperature is 40° C. Aqueous solution of rennin of sp. gr. 1·010 and containing 0·20 per cent. by volume of hydrochloric acid lost 30 per cent. of its activity in twenty-four hours at 21° C. Stronger solutions with more acid proved more permanent. The author's results point to the use of granular scale rennin and phosphoric acid in place of lactic acid in the preparation of essence of pepsin.

H. F. E. H.

Trommer's Test for Sugar in Urine. E. Salkowski. (*Zeitsch. physiol. Chem.*, 1912, 79, 164-170.)—The author agrees with the statements by Schulz (*cf.* ANALYST, 1912, 264) regarding the behaviour of urine when treated with copper sulphate and sodium hydroxide solutions, but points out that urine containing sugar behaves somewhat differently from urine free from sugar when the two are treated with copper sulphate and then rendered alkaline, and that it is still possible to detect sugar in urine by Trommer's test when the order of adding the reagents is reversed. The following manner of carrying out the test is, however, recommended: Five c.c. of a 30 per cent. sodium hydroxide solution and 8 drops of a 10 per cent. copper sulphate solution are mixed and heated to boiling; a clear blue solution is thus obtained containing a few particles of black copper oxide which do not interfere with the test. To the hot solution are now added 2 c.c. of the urine, and the mixture is again boiled. Urine containing as little as 0·2 per cent. of sugar reduces the copper much more than does urine free from sugar. The test is rendered more sensitive by allowing hydrochloric acid to flow on the surface of the hot mixture; in the presence of sugar a yellow zone of hydrated cuprous oxide is obtained.

The urine may also be clarified by means of mercuric acetate before being tested. Twenty c.c. of the sample are treated with a 20 per cent. mercuric acetate solution, this being added drop by drop as long as a precipitate is formed, but a large excess is to be avoided. The mixture is then neutralised with sodium carbonate, filtered, the filtrate acidified with hydrochloric acid and warmed gently after the addition of a quantity of zinc-dust. When all the mercury has been precipitated, the mixture is again filtered, and the filtrate tested for sugar by Trommer's test, the sodium hydroxide being added before the copper sulphate. In the case of normal urine the solution retains its blue colour, but becomes bright yellow if 0·2 per cent. of sugar is present. Subsequent addition of hydrochloric acid also renders this test more sensitive. W. P. S.

ORGANIC ANALYSIS.

Colour Reaction of Alcohols and Alcoholic Hydroxyl Groups. L. Rosenthaler. (*Chem. Zeit.*, 1912, **36**, 830.)—A red coloration is obtained when an alcohol is heated with its own volume of a 0·5 per cent. sodium nitrite solution and 4 volumes of a 0·5 per cent. sulphanilic acid solution (prepared by dissolving 1 grm. of sulphanilic acid in a mixture of 5N hydrochloric acid and 150 grms. of water). With 0·25 grm. of ethyl alcohol or glycerol, the colour develops within thirty seconds; it fades after a time and is discharged on the addition of an acid. The reaction is not very sensitive, but may be obtained with 2·5 mgrms. of ethyl alcohol or 0·01 grm. of glycerol. The coloration develops slowly at the ordinary temperature; this is especially the case with malic acid, whilst citric and tartaric acid yield but a slight reaction even when the mixture is heated. Other acids containing a hydroxyl group or groups give the coloration, as do also the higher alcohols, certain carbohydrates, etc. W. P. S.

Differentiation between Natural and Petroleum-Residue Asphalts. J. Marcusson. (*Chem. Zeit.*, 1912, **36**, 801-803.)—As an approximate means for differentiating between natural and petroleum-residue asphalts, the proportion and nature of the oily constituents afford a guide, but is not in itself a conclusive test. If an asphalt on distillation yields over 34 per cent. of oil of a thick, ointment-like consistence, it is probably a petroleum residue; the oily matters from natural asphalt are relatively thin, but it must be borne in mind that many petroleum residues yield less than 34 per cent. of oil. Further, if the oil contains over 2 per cent. of paraffin-wax, the presence of petroleum residues is very probable, but certain petroleum residues yield oils poor in paraffin—*e.g.*, from Roumanian petroleum 3·1 to 3·6 per cent. and from Californian 2·2 to 2·9 per cent. Further methods of differentiation have been investigated by the author. For instance, natural asphalts, on distillation, yield distillates with a comparatively high acid value. A quantity of the sample representing 30 grms. of bitumen is distilled in a small retort and two fractions of 4 to 5 c.c. each are collected. These are dissolved in ether and shaken with water to remove any mineral acid. Neutral alcohol is then added and the acid value of both fractions determined, using alkali-blue as indicator. In the case of natural asphalt the acid value of the first fraction is over 1, and the second fraction still shows a distinct acid value, whereas the acidity of the distillates from petroleum residues is very small, generally *nil* in the second fraction. The separation of solid paraffin from the distillates is also an indication of petroleum residues. A further criterion is the saponification value of the bitumen soluble in benzene: 5 grms. of the soluble bitumen are dissolved in 25 c.c. of benzene free from thiophene and boiled for an hour with 25 c.c. of $\frac{N}{1}$-alcoholic potassium hydroxide, cooled, diluted with 200 c.c. of alcohol and titrated with repeated heating until all the alkali is extracted from the precipitate of pitch. Natural asphalts show saponification values of 28·5 to 34 for the extracted bitumen; petroleum residues show values from 7·6 to 13·5. J. F. B.

Bromine and Iodine Methods for the Volumetric Estimation of Cresol. C. M. Pence. (*J. Ind. and Eng. Chem.*, 1912, **4**, 518-520.)—The usual bromine methods for the estimation of cresol depend upon fixed conditions (*cf.* ANALYST, 1900, **25**, 78), and whilst they may be of service they do not satisfy the demand for a suitable method. Tribromo-*o*- and *p*-cresol bromides cannot be formed in a manner analogous to the production of tribromophenol bromide (*cf.* ANALYST, 1910, **35**, 218); dibromocresol bromide compounds are formed and finally tribromophenol bromide, but *o*- and *p*-cresols cannot be estimated volumetrically by conversion into these compounds. *m*-cresol may be estimated by conversion into tribromo-*m*-cresol; if the *m*-cresol solution is in contact with bromine for 1·5 hours and potassium iodide is then added and the mixture allowed to stand for two hours, all the cresol is converted into tribromo-*m*-cresol.

O- and *p*-cresols form di-iodo compounds, and this reaction is made the basis of a method for their estimation. Five grms. of the cresol are dissolved in water, 10 c.c. of $\frac{N}{2}$ sodium hydroxide solution being added to aid in the solution, and the mixture is diluted to 1 litre. Twenty-five c.c. of this solution are treated with 9 grms. of sodium acetate and 50 c.c. of $\frac{N}{10}$ iodine solution. After the lapse of one hour, 100 c.c. of water are added and a few c.c. of chloroform to dissolve the precipitated di-iodocresol, and the excess of iodine is titrated with thiosulphate solution. As *m*-cresol does not yield di-iodo compounds under the same conditions, a mixture containing this cresol cannot be analysed by a volumetric iodine method. W. P. S.

Compounds of Ferric Salts with Albumoses. F. Rohmann and T. Shmamine. (*Biochem. Zeitsch.*, 1912, **42**, 250-254.)—When an aqueous solution of Witte's peptone is treated with either ferric chloride or ferric sulphate, a precipitate containing primary albumoses is obtained, which may be collected on a filter and washed without difficulty. It contains sulphuric acid in addition to iron, when ferric sulphate is used, but the ratio of Fe : SO_4 is greater than in the precipitant. When the precipitate is suspended in water and an alkali is added cautiously, complete solution is obtained, but on the addition of an excess of alkali ferric hydroxide is precipitated and the albumoses remain in solution. If barium hydroxide be used as the alkali, the sulphuric acid is also precipitated. After removing the excess of barium hydroxide by means of carbon dioxide, the barium-albumose compound may be decomposed with dilute sulphuric acid; a portion of the albumoses remains in solution whilst the remainder separates out together with the barium sulphate. The insoluble portion is soluble both in alkali solutions and in very dilute sulphuric acid. This behaviour of the albumoses recovered from the iron precipitate indicates the presence of a mixture of proto- and hetero-albumose. W. P. S.

Estimation of Lactic Acid and β-Hydroxybutyric Acid in Mixtures of the Same. J. Mondschein. (*Biochem. Zeitsch.*, 1912, 42, 91-104.)—When lactic acid in 1 per cent. sulphuric acid is heated to boiling and $\frac{N}{10}$ potassium permanganate solution is added drop by drop, the lactic acid is converted quantitatively into acetaldehyde, carbon dioxide, and water, according to the equation :

$$CH_3.CHOH.COOH + O = CH_3.CHO + CO_2 + H_2O.$$

On the other hand, β-hydroxybutyric acid in 4 per cent. sulphuric acid solution is converted into acetone, carbon dioxide, and water, by adding to the boiling solution a 0·5 per cent. potassium bichromate solution; in this case the reaction proceeds according to the equation :

$$CH_3.CHOH.CH_2.COOH + O = CH_3.CO.CH_2.COOH + H_2O = CH_3.CO.CH_3 + CO_2 + H_2O,$$

the acetoacetic acid being an intermediate product. Should both lactic acid and β-hydroxybutyric acid be present together in a solution, the former yields a small quantity of aldehyde when the mixture is oxidised with bichromate, and the latter acid a small quantity of acetone during the permanganate oxidation, the conditions necessary for the complete oxidation of one acid not being suitable for the complete decomposition of the other. The two acids may, however, be estimated separately by a combination of these methods, the oxidations being carried out in an apparatus described previously by von Furth and Charnass (ANALYST, 1910, 35, 445). A definite volume of the solution containing the two acids is oxidised with permanganate as described (*loc. cit.*), and the distillate obtained, measuring 400 c.c., is divided into two equal portions, and the total aldehydic content is estimated in one portion by means of potassium bisulphite as described by Ritter (ANALYST, 1901, 26, 131). The other portion of the distillate is then boiled under a reflux condenser after the addition of hydrogen peroxide and sodium hydroxide in order to destroy the aldehyde. The mixture is now distilled, and the distillate again examined by Ritter's method. The difference between the results of the two estimations corresponds with the amount of aldehyde present, from which the lactic acid may be calculated.

Another portion of the original solution is then oxidised with potassium bichromate, and distilled. The distillate in this case contains the whole of the acetone derived from the β-hydroxybutyric acid together with a quantity of aldehyde from the lactic acid. After this aldehyde has been destroyed by boiling the solution with sodium hydroxide and hydrogen peroxide under a reflux condenser, the solution is again distilled and the acetone estimated in the distillate by Messinger's iodoform method (*cf.* ANALYST, 1908, 33, 98). Experiments with solutions containing known quantities (about 0·2 grm. each) of the two acids show that the method is trustworthy, 99·2 per cent. of the amount lactic acid, and 97·8 per cent. of the β-hydroxybutyric acid actually present being obtained. W. P. S.

Method of Examining Chinese Wood Oil. P. C. McIlhiney. (*J. Ind. and Eng. Chem.*, 1912, **4**, 497-498.)—The following method of estimating the proportion of this oil which is solidified when treated with iodine is proposed in the hope that the estimation may be of value in distinguishing between genuine oils and those which have been adulterated with other oils. The author refrains from giving any figures at present, as he has not been able to obtain authenticated specimens of Chinese wood oil, and has only applied the method to commercial samples. Broadly speaking, Chinese wood oil yields only a small fraction of liquid oil when subjected to the process, whilst other oils remain entirely liquid and soluble in petroleum spirit. The details of the method are as follows: Five grms. of the oil are heated with 10 c.c. of 99·5 per cent. acetic acid until a clear solution is obtained, and 50 c.c. of a 1·5 per cent. iodine solution in 99·5 per cent. acetic acid are added. This iodine solution should be warmed so that it does not cool the oil solution. After the lapse of thirty minutes, 50 c.c. of petroleum spirit are added, mixed thoroughly with the mass, and then decanted into a separating funnel; the extraction with petroleum spirit is twice repeated, and the united extracts are washed with water until free from acid, then with potassium iodide solution to remove free iodine, and finally with water. The petroleum spirit is next evaporated and the residue weighed in order to obtain the portion of the oil which is not solidified by the action of iodine.

W. P. S.

Heat Test for Chinese Wood Oil. F. Browne. (*Chem. News*, 1912, **106**, 14-15.)—The author advocates a standardised form of apparatus and method of working for the observation of the time required to produce "setting" or jelly formation on heating the oil to 540° to 560° F. The addition of 10 per cent. of other vegetable, or of mineral, oils causes an appreciable lengthening of the time required, which for 5 c.c. of a genuine wood oil is about eleven to twelve and a half minutes under the stated conditions of the experiments. If the time required is thirteen minutes or more, the author employs the iodine and chloroform test (ANALYST, 1898, **23**, 43). Genuine oils "set" in from forty to fifty-five seconds. The following range of constants is given: Sp. gr. at 15·5° C., 0·9402 to 0·9431; saponification number, 191 to 194; iodine number (Wijs), 173 to 174. A. R. T.

Refractive Index of Chinese Wood Oil. L. E. Wise. (*J. Ind. and Eng. Chem.*, 1912, **4**, 497-498.)—The refractive indices of fifteen samples of commercial Chinese wood oil were found to lie between 1·5099 and 1·5186, the values being determined at a temperature of 25° C. Gross adulteration of the oil with other drying oils can be detected readily by means of the refractometer as the refractive index is lowered. W. P. S.

Effect of Free Fatty Acid on Specific Gravity of Some Fatty Oils. A. O. Ransome. (*J. Soc. Chem. Ind.*, 1912, **31**, 672.)—The author's results support the statement of Allen ("Commercial Organic Analysis," II., part i., pp. 126-127) that in the case of olive oil the presence of free fatty acids lowers the sp. gr. Each 5 per cent. of free acid was found by the author to diminish the gravity by

0·00082, Allen's figure being "about 0·0007." From a consideration of the results of other observers, the author shows that the sp. gr. of the fatty acids of various oils is invariably lower than that of the oil itself, the differences being from 0·0037 to 0·0258. It is further shown that the iodine number of Menhaden oil is increased by the presence of free fatty acids, and it is assumed that many or all constants of an oil will vary progressively with the proportion of free acids present. In the case of oils containing a sufficient quantity of free acids to vitiate the constants, it is thus considered desirable to isolate the fatty acids and make the analysis on them instead of on the original oil.

The following figures may be quoted:

ANALYSIS OF SIX AUTHENTIC SPECIMENS OF OLIVE OIL.

	Sp. Gr., 15·5° C.	Neutral Sp. Gr.	Free Acid as Oleic, per Cent.	Decrease due to 5 per Cent. Free Acid.
Malaga, No. 1	0·91572	—	2·24	—
„ No. 2	0·91554	—	2·79	—
„ No. 5	0·91528	—	3·22	—
Calabria, fino	0·91654	—	1·38	—
„ aino-verde ...	0·91374	0·91800	25·87	0·000820
„ aino-giallo ...	0·91280	0·91872	36·38	0·000825

ANALYSIS OF A SAMPLE OF MENHADEN OIL CONTAINING 4·03 PER CENT. GLYCEROL.*

	Sample.	Glycerides.	Acid.	Corrected Acids.
Sp. gr. at 15·5° C. ...	0·9257	0·9309	0·9161	0·9149
Iodine value (Wijs) ...	185·27	180·39	192·02	192·9
Free fatty acids, per cent. (= oleic)	47·99	0·22	—	—
Acid value	—	—	187·42	—
Saponification value ...	—	192·9	200·80	—

Three Menhaden oils showed a decrease of sp. gr., for each 5 per cent. of free fatty acid, of 0·00054, 0·00084, and 0·00063. A. R. T.

Glycerides Occurring in Fats and Oils: Heptadecoic Acid and its Glyceride. A. Bömer and R. Limprich. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, 23, 641-653.)—Considerable doubt having arisen as to the actual presence of a glyceride of heptadecoic acid in fats and oils (*cf.* ANALYST, 1904, 29, 259; 1905, 30,

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* The acids contained about 7 per cent. of glyceride calculated on the difference between the neutralisation and saponification values. The figures for acids were corrected on this basis.

212), the authors have prepared this acid and its glyceride synthetically for the purpose of comparison with the acid isolated from fats and oils; the results of this comparison will be published later, the present paper dealing with the preparation and properties of the acid. Krafft's method (*Ber.*, 1879, **12**, 1668) was used for this purpose; it consists in distilling a mixture of barium stearate and barium acetate under reduced pressure, separating the resulting ketones by fractional solution in alcohol, oxidising the heptadecyl ketone with bichromate and sulphuric acid, and crystallising the heptadecoic acid repeatedly from alcohol. The pure acid obtained melted at 60·5° C., and boiled, in an absolute vacuum, at 143·6° C. One hundred c.c. of absolute alcohol dissolve 1·15 grms. of the acid at 0° C., and 3·48 grms. at 15° C. The zinc salt is practically insoluble in cold alcohol. The triglyceride of the acid was prepared by heating the potassium soap for two hours with the theoretical quantity of tribromhydrin, extracting the mass with ether and evaporating the ethereal solution; the residue obtained was dissolved in benzene, and the glyceride precipitated by the addition of alcohol. After crystallisation from ether it yielded masses of needle-shaped crystals which melted at 62·7° C. One hundred c.c. of anhydrous ether dissolve 0·0288 grm. of the glyceride at 0° C., and 0·322 grm. at 15° C. W. P. S.

New Oxygen Absorption Method for Oils. L. P. Wilson and G. S. Heaven. (*J. Soc. Chem. Ind.*, 1912, **31**, 565-568.)—About 0·2 grm. of the oil is mixed in a flask with 1 grm. of kieselguhr, and, after the flask has been cooled to a definite temperature, a side-tube provided on the neck of the flask is sealed. The flask is now heated to 100° C. for one hour, again cooled, and the side-tube is connected with graduated levelling tubes filled with water; the height of the water is noted and the seal is broken, the resulting difference in the water-level (after the water surfaces have been brought to the same level) being a measure of the amount of oxygen absorbed by the oil. The volume of oxygen absorbed is then calculated into a percentage quantity on the oil. The side-tube of the flask may be provided with a bulb to prevent water entering the flask. The following percentage amounts of oxygen were absorbed by various oils examined: Linseed oil, 17·2 to 21·2; poppyseed oil, 17·4; cottonseed oil, 11·1; sesame oil, 5·8; olive oil, none. W. P. S.

Notes on Serger's Reaction for Vegetable Oils. Utz. (*Chem. Rev. Fett.- u. Harz.-Ind.*, 1912, **19**, 128-131; 151-153.)—Serger has suggested the use of a solution of 0·1 grm. of sodium molybdate in 10 c.c. of strong sulphuric acid as a means of distinguishing between vegetable and animal oils, the former when dissolved in ether giving colorations varying from yellow to blue or green, whilst in the case of the latter no colorations are produced in less than thirty to forty-five minutes. Comparative tests here described show that the reaction is slower and no more sensitive than Bellier's test, whilst treatment of the oils with bleaching agents such as benzoyl peroxide ("lucidol") or the action of heat prevent the appearance of the colorations. Oils giving only faint reactions in this test may be mixed with lard without risk of detection by Serger's test, and the phytosterol test is then the only method of proving the addition of vegetable oils. C. A. M.

Comparison of Methods for Determining the Viscosity of Pitch. B. Weinberg and H. Smirnow. (*J. Russ. Phys.-Chem. Soc.*, 1912, **44**, 3; through *Chem. Zentralbl.*, 1912, II., 4.)—The investigation was confined to methods in which the motion reaches a limit, and in which alteration of form, but not of volume, takes place (*cf.* Weinberg, *J. Russ. Phys.-Chem. Soc.*, 1907, **38**, 202). The torsion method, the Legel method, and the strata-displacement method, were particularly examined. The torsion method is the best for substances having viscosities of the order of $10^8 \frac{\text{g.}}{\text{cm. sec.}}$; it may be used for substances from 10^6 to 10^{10}, but the external friction of the apparatus must be accurately determined. The Legel method possesses the advantage of direct transmission of the force to the examined substance, but secondary phenomena must again be taken into account. The third method has a low degree of accuracy, and for substances of viscosity below 10^9 requires the application of the law of Archimedes; this applies also to the first method when the viscosity is below $10^{10} \frac{\text{g.}}{\text{cm. sec.}}$ O. E. M.

Separation and Estimation of Pyridine and Ammonia. A. Bayer. *J. f. Gasbeleuchtung*, 1912, **55**, 513-514; through *Chem. Zentralbl.*, 1912, II., 281.)—In the titration of pyridine and ammonia, ferric thiocyanate is a better indicator than methyl orange or ferric chloride. The solution of pyridine or ammonia is acidified with $\frac{N}{10}$ hydrochloric acid, ferric chloride and ammonium thiocyanate are added, and then $\frac{N}{10}$ caustic alkali, until the red-brown liquid becomes quite colourless. For the estimation of pyridine in presence of ammonia, the pyridine is first concentrated by distilling the solution after the addition of sulphuric acid until it is almost neutral to litmus. The whole of the pyridine, with very little ammonia, passes over with the first third of the distillate. The ammonia is titrated with $\frac{N}{10}$ hydrochloric acid, using litmus as indicator, excess of acid is then added, and the pyridine estimated in the manner already described. Where the utmost accuracy is desired, the ammonia is best eliminated as ammonium magnesium phosphate.

G. C. J.

Detection of Pyridine Bases in Ammonia and Ammonium Salts. A. Wöhlk. *Ber. deutsch. pharm. Ges.*, 1912, **22**, 285-288; through *Chem. Zentralbl.*, 1912, II., 286-287.)—If an ammonium salt containing pyridine bases be triturated in a mortar with twice its bulk of borax, the impurity may be detected by its odour, which is pronounced if so little as 0·4 mgrm. be present. For the examination of liquid ammonia, the latter is mixed with a small excess of acid and evaporated to dryness on the water-bath, the dry residue being used for the test. Ammonium salts can be freed from pyridine by dissolving them in water and leading a current of pure ammonia gas through the solution. G. C. J.

A Critical Study of Sugar Analysis by Copper Reduction Methods. A. W. Peters. (*J. Amer. Chem. Soc.*, 1912, **34**, 928-954.)—The investigation described by the author arose from the need for a rapid and accurate method of determining sugar in urine, and led to his standardising a volumetric method applicable to any sugar. No new principle is involved, the experimental work recorded mainly dealing with the effect produced by variations in the rate of heating the alkaline copper solution with sugar. To an aliquot portion of the residual copper solution after filtration sulphuric acid and excess of potassium iodide are added, and the liberated iodine titrated against thiosulphate. This "residual" method the author considers very rapid and accurate. An examination in detail is described of the rate of reduction with pure dextrose under varying conditions of time and heating, and curves are given showing the percentage of the total sugar reduced for each interval of time at different temperatures. It is important to employ a mechanical heating device to insure uniformity in this connection, and the standard heating power arbitrarily selected was such as to produce a rise from 35° to 90° C. in 120 seconds, when acting on 60 c.c. of distilled water. The duration of heating was always calculated from the moment the temperature of 90° C. was reached, and was fixed at exactly 20 seconds. Reasons are given and figures quoted supporting the contention that this is the best length of time for the reduction process, the much longer period usually taken being without justification. The total volume of the solution employed is 60 c.c., 20 c.c. of "Fehling-Soxhlet" copper sulphate solution, 20 c.c. of "Allihn alkaline tartrate solution," containing 346 grms. of Rochelle salt and 250 grms. caustic potash per litre, and 20 c.c. of sugar solution. The heating is conducted in an Erlenmeyer flask, closed with a doubly-bored stopper, a thermometer passing through one of the holes, the other being left free for the escape of steam. The flask stands on an asbestos plate, which must be 3 to 5 cm. away from the burner. At the end of the 20 seconds' heating, the whole is cooled for a few seconds under the tap to just below the boiling-point, and then filtered through a Gooch crucible packed very heavily with two layers of asbestos containing pure talcum powder between them. The total volume of filtrate, including washings, must not exceed 75 to 80 c.c.; and the thiosulphate finally employed should have a value between 12·5 and 13 mgrms. of copper per c.c. The dextrose-copper ratio so obtained falls from 0·833, when 1 mgrm. of dextrose is employed, to 0·522 for 25 mgrms. dextrose, at which point it remains constant till 110 mgrms. of dextrose is reached, when it rises to 0·572 at 200 mgrms. dextrose. From 1 to 10 c.c. of urine may be employed, 2 c.c. being usually taken in the case of urine from diabetic subjects.

H. F. E. H.

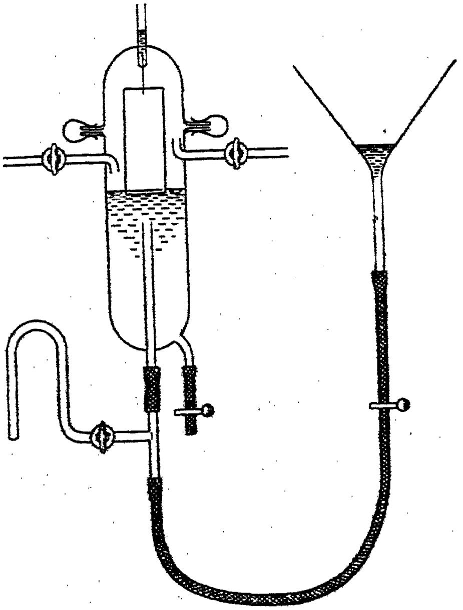
INORGANIC ANALYSIS.

Separation of Arsenic from Antimony and Other Metals by Means of Methyl Alcohol in a Current of Air. L. Moser and F. Perjatel. (*Monatsh.*, 1912, 33, 797-820.)—The authors have worked out a method for the estimation of arsenic by distillation in presence of methyl alcohol and strong hydrochloric acid with a current of air at the temperature of the water-bath (*cf.* Friedheim and Michaelis, *Ber.*, 1895, **28**, 1414; S. W. Collins, ANALYST, 1912, 229). A portion of the arsenic distils as the trichloride, and a portion as methyl arsenite, which is immediately hydrolysed by the water in the receiver. If the arsenic is present in the pentavalent form, it must be reduced before distillation, preferably by means of a ferrous salt. The methyl alcohol has no reducing action under the conditions adopted. The usual method of oxidising the arsenious acid, in preparing ores for analysis, by potassium chlorate and hydrochloric acid is unsatisfactory, and leads to a loss of arsenic; but oxidation by means of chlorine in alkaline solution is complete. Arsenic acid is not volatilised with hydrochloric acid. The general arrangement of the apparatus used for distillation is similar to that described by Thaddeeff for the estimation of boric acid with methyl alcohol. It consists of a wide-necked flask, with rubber stopper having three holes. Through one passes a tube for the introduction of air, extending to some height; through the other a dropping funnel; and through the third a simple bulb distillation-head. To this is joined a tube 60 cm. long, bent at one end, and dipping to the bottom of a beaker cooled by water. The flask is immersed in a water-bath; the current of air is regulated by a screw-clip, and passes through drying towers. About 200 c.c. of water are placed in the beaker. For the separation of arsenic from antimony, etc., the quantity of arsenious acid taken must not exceed 0·22 grm. If the substance has to undergo a preliminary treatment, this may be carried out in the flask, and any residues of nitric acid or water must be removed; small quantities of sulphuric acid are immaterial. In the case of pentavalent arsenic, 5 to 8 grms. of ferrous sulphate are added; then 40 to 50 c.c. of hydrochloric acid, sp. gr. 1·19, and the contents of the flask are brought to the boil in the water-bath, and kept at the boil for five to ten minutes. By that time the reduction of the arsenic acid is complete, and the first addition of 30 c.c. of methyl alcohol is made, air being passed through for a few seconds to prevent the sucking back of water in the beaker. The liquid is then boiled for a quarter of an hour without the air-current, and for half an hour with a vigorous current. Next, a further 20 c.c. of methyl alcohol are added, and boiled for a quarter of an hour; the same operation is repeated, and the liquid boiled for another quarter of an hour. The total time of boiling is one and a half hours, of which one hour is with the air-current. The separation of the arsenic is complete, and this element is estimated in the distillate either gravimetrically or volumetrically by iodine or permanganate. Th antimony and other metals are estimated in the residue; the method is not applicable for the separation of arsenic from mercury, since the latter is also volatile as chloride.

J. F. B.

Estimation of Arsenic. E. Rupp and F. Lehmann. (*Arch. Pharm.*, 1912, **250**, 382-389.)—The following method has been devised more especially for the rapid estimation of arsenic from salvarsan in animal tissue. It depends upon the destruction of the organic matter by means of potassium permanganate or persulphate and sulphuric acid, and the distillation of the arsenic as trichloride in a current of hydrochloric acid gas evolved from the solution itself. From 5 to 20 grms. of divided moist material are intimately mixed with 10 grms. of powdered potassium permanganate (or persulphate) and 10 c.c. of dilute sulphuric acid, and the mixture heated for fifteen minutes, with continual stirring, on the water-bath, after which it is gradually treated with 25 c.c. of concentrated sulphuric acid, followed by 30 c.c. of hydrogen peroxide solution. As soon as frothing has stopped, the liquid is transferred to a Kjeldahl flask, into which is also introduced 5 grms. of anhydrous (or 10 grms of crystalline) ferrous sulphate to reduce the arsenic acid, and 50 grms. of sodium chloride, the flask then connected with a receiver containing 40 grms. of sodium bicarbonate and 100 c.c. of water, and heated on a sand-bath. The receiver is frequently shaken, and as soon as the whole of the bicarbonate has dissolved the distillation is stopped, and the distillate cooled, rendered alkaline, if necessary, with sodium bicarbonate, filtered, and titrated with standard iodine solution, 0·05 c.c. of $\frac{N}{10}$ solution being deducted as a correction. C. A. M.

Electrometric Determination of the Reaction of Liquids containing Carbon Dioxide. K. A. Hasselbalch. (*Compt.-rend. des Travaux du Lab. de Carlsberg*, 1911, **10**, 69-84.)—The exact electrometric determination of the reaction of liquids containing carbon dioxide is rendered difficult by the tendency of the solution to part with some of its dissolved gas, and become appreciably more alkaline before the determination can be completed. Michaelis and Rona (*Biochem. Zeitsch.*, 1909, **18**, 317) have proposed to obviate this difficulty by superimposing an atmosphere of hydrogen above the liquid, and by preventing the electrode from dipping in the liquid any more than is necessary to make contact. Their method has advantages, but does not give exact results, and it is now shown that an atmosphere of hydrogen does not prevent loss of carbon dioxide, and that the increased accuracy of their results compared with those of earlier workers is entirely due to the electrode not being submerged in the liquid.



The author has adopted this principle of slight immersion of the electrode, and has evolved a method which makes it possible to obtain exact results in a very short time (half an hour). The method depends on putting some of the solution in equilibrium with hydrogen in an appropriate apparatus, and then, since the liquid has lost carbon dioxide in the process, replacing it by a fresh portion of liquid, and proceeding to the electrometric measurement. The illustration shows a convenient form of apparatus. The electrode is seen just touching the surface of the liquid, which half fills a vessel of about 15 c.c. capacity. A current of hydrogen is passed through the empty vessel for half an hour to saturate the hydrogen electrode at the prevailing barometric pressure. The liquid is then introduced through the funnel and pinchcock, and the apparatus rocked from the vertical to a horizontal position and back again some 200 times. Blood may require nearly three times as much agitation. The liquid, which has lost carbon dioxide, is now run out of the apparatus and replaced by a fresh portion, and the E.M.F. determined in the usual manner.

G. C. J.

Estimation of Arsenious Acid with Permanganate in Presence of Hydrochloric Acid. L. Moser and F. Perjatel. (*Monatsh.*, 1912, **33**, 751-758.) —The titration of arsenious acid by permanganate in acid solution sometimes presents difficulties, owing to the fact that reduction to the manganous stage is only complete and rapid under certain conditions. If the permanganate be added too quickly to a too concentrated solution of arsenic, yellow to brown colorations, due to the intermediate stages of reduction of the manganese, occur, and the titration is spoilt; the concentration of free acid is also an important factor. The authors have worked out the following generally satisfactory method: 0·1 to 0·3 grm. of arsenious acid is dissolved in not very concentrated caustic soda lye, containing about 1 grm. of solid sodium hydroxide, in a porcelain dish, and the solution is diluted with 100 to 200 c.c. of water. The liquid is acidified by the addition of 10 to 15 c.c. of hydrochloric acid, sp. gr. 1·19, per 1 grm. of sodium hydroxide used. Permanganate is then added drop by drop in the cold with continuous stirring. Under these conditions no brown coloration ensues, and the end-point is sharply recognisable by a pink colour lasting for two to three minutes. The volume of the arsenious chloride solution should be between 50 and 200 c.c. If the solution to be tested is already acid, it is advisable to neutralise it first exactly with sodium hydroxide, and then to add 5 to 10 c.c. of concentrated hydrochloric acid. If a yellow coloration develops on titration, the addition of the permanganate has been too rapid. The results are accurate.

J. F. B.

Reaction of Hydrazine and Hydroxylamine with Ferricyanides, and New Methods for Estimating Hydrazine and Ferricyanides. P. R. Rây and H. K. Sen. (*Zeitsch. anorg. Chem.*, 1912, **76**, 380-386.)—Hydrazine and potassium ferricyanide in alkaline solution react as follows:

$$N_2H_4 + 4K_3Fe(CN)_6 + 4KOH = N_2 + 4K_4Fe(CN)_6 + 4H_2O.$$

In presence of excess of alkali, the reaction is quantitative until either the whole of the hydrazine or the whole of the ferricyanide has disappeared. The reaction proceeds rapidly at room-temperatures, and is unaccompanied by any secondary reactions. It therefore affords a basis for the estimation of hydrazine or ferricyanide.

For the estimation of hydrazine, the substance for analysis is weighed out from a weighing bottle into the cup of a Crum's nitrometer, and washed into the latter by means of a little water and 4 to 5 c.c. of 15 per cent. caustic potash. A solution of the substance may be used, since considerable dilution has no influence on the course of the reaction; but the presence of a deep aqueous layer above the mercury in the nitrometer reduces the space available for gas, and the larger the gas volume, the less the percentage error of the method. A large crystal (an excess) of potassium ferricyanide is introduced by the open end of the nitrometer, which is immersed in the mercury bath, and the tube is shaken to hasten the reaction, which is complete in three or four minutes.

Since Stolle's iodine method (*J. Prakt. Chem.*, 1902, **66**, 332) for the estimation of hydrazine leaves nothing to be desired, the new method is recommended rather for the estimation of ferricyanide, especially in presence of ferrocyanide. In this application, a "pill" of hydrazine sulphate (an excess) is introduced into the nitrometer from below, and the ferricyanide is weighed out into the cup of the nitrometer, covered with water, and drawn in. Caustic potash solution is then added through the cup, and the analysis completed as before. The accuracy of the method is limited only by the accuracy with which the gas volume can be read. The test numbers show that the authors make no attempt to read this volume more closely than 0·05 c.c., and their greatest error corresponds exactly to a misreading of the gas volume of 0·05 c.c. The excess of hydrazine always present in ferricyanide determinations keeps the mercury clean, but mercury which has been exposed to excess of ferricyanide in hydrazine determinations needs purification with dilute nitric acid before being used for other purposes.

Phenyl-hydrazine reacts with ferricyanide similarly to hydrazine, but hydroxylamine gives rise to a mixture of nitrogen, ammonia, and nitrous oxide, the proportions of which depend on the exact conditions under which the reaction is brought about. G. C. J.

Determination of Iodine in the Presence of Other Halogens and Organic Matter. E. C. Kendall. (*J. Amer. Chem. Soc.*, 1912, **34**, 894-909.)—For the determination of iodine, when present as a soluble iodide or in the uncombined form, the author employs a modification of Hunter's method (Analyst, 1910, **35**, 483), in which about 200 c.c. of the cold neutral or slightly alkaline solution, containing the iodine to be estimated, is treated slowly with sodium hypochlorite in slight excess in the presence of 5 c.c. of 42 per cent. phosphoric acid. Any iodine present is oxidised to iodic acid. To prevent loss of iodine, if more than 0·1 grm. is present, a few c.c. of benzene are floated on the surface of the liquid. Ten c.c. of a colourless 5 per cent. solution of phenol are then rapidly and forcibly blown into the flask. This removes all traces of chlorine without the formation of coloured compounds. The whole is now made just alkaline with 30 per cent. sodium hydroxide, and then acidified by the addition of 10 c.c. of 50 per cent. phosphoric acid. Potassium iodide is next added in excess, about eight times the weight of the iodine originally present being required; and the liberated iodine, of which one-sixth part is that originally present, is titrated with thiosulphate. Working with quantities of iodine lying between 5 and 350 mgrms., over fifty results are recorded, showing a maximum error of −0·76 per cent., a minimum error of +0·02 per cent., and an average error of about ±0·1 per cent.

Bromine or bromides, if present, do not interfere, and there is no oxidation of the bromine. Hydrobromic acid, when present in large amount, will reduce iodic acid, but all the former may be removed by the addition of sufficient hypochlorite. The procedure is that already described, except that 10 c.c. of benzene are added, sufficient hypochlorite being used to liberate all the bromine and oxidise the iodine.

A little pumice is added, and the solution is boiled. The benzene reacts with the hypobromite and hypochlorite, forming bromo- and chloro-benzene. The free bromine is boiled out of the solution. A precipitate of bromo-benzene may form, but does not interfere. When colourless, the solution is cooled, and potassium iodide is added directly, no neutralisation and acidification being necessary. The presence of chlorides has no effect, and there is no need to boil the solution.

To remove organic matter, enough material to contain about 60 mgrms. of iodine is fused with special precautions, at a temperature below dull redness, in a nickel crucible, with 5 grms. of solid caustic soda. Potassium nitrate is finally added in small quantity, and the melt, which should be clear, is cooled and extracted with water. At this stage, silver, copper, iron, or mercury, if present, are removed, and the solution, which contains sodium hydroxide, carbonate, and iodide, potassium nitrite and nitrate, is acidified slowly with 10 c.c. of 50 per cent. sulphuric acid, after the addition of 2 c.c. of a 10 per cent. solution of sodium bisulphite, to prevent loss of iodine with the escaping carbonic acid, and a few c.c. of chloroform. The iodine, which is now present as hydriodic acid, is oxidised with hypochlorite to iodic acid, as already described, phenol and potassium iodide being added as before. If bromine is present, the modifications required in its presence mentioned above are introduced. The author was unable to modify Hunter's method (*loc. cit.*) satisfactorily for the estimation of iodine in such small quantities as those present in preparations of thyroid gland. A better method is the fusion treatment already described, but with numerous detailed modifications, gallic acid being added after the potassium nitrate to effect the reduction of the excess of nitrate and nitrite. Bromine is next added in slight excess, and then removed with sodium salicylate. When this method is employed with all the refinements described, as little as 0·005 mgrm. of iodine can be detected with certainty, 1 grm. of thyroid gland substance and 10 grms. of caustic soda being suitable amounts to employ for the preliminary fusion. Three commercial samples of thyroid gland preparation contained respectively 1·899, 1·950, and 1·716 mgrms. of iodine per grm. of material.

H. F. E. H.

Electrolytic Estimation of Manganese and its Separation from Iron. H. Golblum and H. Gunther. (*Compt. rend.*, 1912, 155, 166-168.)—The presence of iron in a solution from which manganese is to be deposited as MnO_2 is a positive advantage, as the deposit is then much more adherent. In solutions containing iron and manganese, the latter may be deposited completely as MnO_2 as follows: The solution, which may conveniently contain 0·1 grm. of manganese and 10 grms. of ammonium oxalate in 100 c.c., is placed in a platinum Classen dish, which serves as an anode, the rotating cathode consisting of a smaller slotted dish suspended from the rotating device by a platiniridium rod of sufficient rigidity. The cathode is rotated at about 700 revolutions per minute, and a current of 2 ampères at 3 volts employed, the solution being maintained at 80° C. An hour suffices for the deposition of the whole of the manganese as MnO_2 on the anode, and, under the above conditions, the deposit will contain not more than 0·5 mgrm. nor less than 0·2 mgrm. of iron. The exact correction corresponding to any particular conditions may be readily determined by solution of the weighed deposit and colorimetric determination of the iron. Lower current densities reduce the amount of iron deposited on the anode, but a current of at least 0·5 ampère is necessary to make the deposition of manganese quantitative. Some iron, but comparatively little, is deposited on the cathode, which may be weighed, and the solution freed from manganese may then be electrolysed in other suitable apparatus for the estimation of the iron. G. C. J.

Electrolytic Estimation of Molybdenum. Part I. A. Fischer and J. Weise. (*Zeitsch. anal. Chem.*, 1912, **51**, 537-550.)—The authors are unable to confirm the statement of Exner (ANALYST, 1903, **28**, 328) and of Wherry and Smith (*ibid.*, 1907, **32**, 341) that molybdenum is deposited quantitatively from a molybdate solution as hydrated sesquioxide when following the directions of those authors. On the other hand, the quantitative deposition of molybdenum is found to be practicable under the following conditions: When speed is no object, or the complication of a rotating anode is undesirable, the molybdate solution is acidified with 0·15 c.c. sulphuric acid, diluted to 120 c.c. in a platinum dish with matte surface, heated to an initial temperature of 75° C., and electrolysed for fourteen hours with a current of 0·02 to 0·03 ampère at 1·8 to 2 volts. The deposit is converted quantitatively into MoO_3 by heating for two hours in an electrically-heated furnace maintained at 400° C. For rapid work, hydrochloric acid must be substituted for sulphuric acid and, with from 0·05 to 0·2 grm. MoO_3 present, the amount of hydrochloric acid should not be much less than 0·2 c.c., nor more than 0·25 c.c. of concentrated acid (sp. gr. 1·19). If no more than 0·13 c.c. of acid be present, the deposit is not adherent, whilst high concentrations lead to incomplete deposition. With as much as 0·33 c.c. acid present, the results are always low but never more than 0·5 mgrm. low; but two or three times this amount of acid gives rise to errors of from 10 to 50 per cent. A current of 3·5 to 4 ampères is employed, the voltage varying from 18 to 30 according to the temperature of the electrolyte and the speed at which the anode is rotated. A convenient speed is 600 to 800 revolutions. Deposition is complete in less than twenty-five minutes. The deposit is transformed into MoO_3 in the manner already described. A current of 2·5 ampères gives equally good results, but thirty-five minutes are required for the deposition of 0·2 grm. The authors have been unable to get good results when working with larger quantities than correspond to 0·2 grm. MoO_3, and they show that the error depends mainly, if not entirely, on the difficulty of oxidising large deposits quantitatively to MoO_3. The success of this operation, even with small deposits, is shown to depend on precise regulation of the temperature. At temperatures much below 400° C., the conversion of Mo_2O_3 to MoO_3 is never complete, whilst higher temperatures may result in loss by volatilisation. G. C. J.

Analysis of Platinum Alloys. H. Arnold. (*Zeitsch. anal. Chem.*, 1912, **51**, 550-554.)—The analysis of platinum alloys can be accomplished more quickly by the following method than by methods which depend on the separation of platinum as metal or as sulphide. The method depends on the separation of platinum as potassium platinic chloride. The alloy (1 grm.) is dissolved in *aqua regia*, and the solution evaporated to dryness three times with hydrochloric acid. The soluble chlorides are taken up in 10 c.c. of hydrochloric acid (sp. gr. 1·12), 1 grm. of caustic potash dissolved in a little water is added and the solution evaporated to dryness on a not too vigorously boiling water-bath. The residue of silver chloride and potassium platinic chloride is washed four or five times by decantation with 50 c.c. of alcohol, and, the bulk of the precipitate is dried in the evaporating basin whilst the filter is ashed. The dried precipitate is next transferred to the crucible containing the ash, and the potassium platinic chloride decomposed by ignition in a current of hydrogen. The contents of the crucible are then transferred to a beaker and heated successively with dilute nitric acid and dilute ammonia. The platinum is filtered off, washed with hot dilute ammonia and then with hot water, and the filter ashed and the residue ignited, treated with hydrofluoric acid to eliminate silica derived from glass vessels, re-ignited and weighed. In the filtrate from platinum, silver is precipitated —preferably as sulphide, since silver chloride is appreciably soluble in solutions of alkali chlorides—and is finally weighed as chloride.

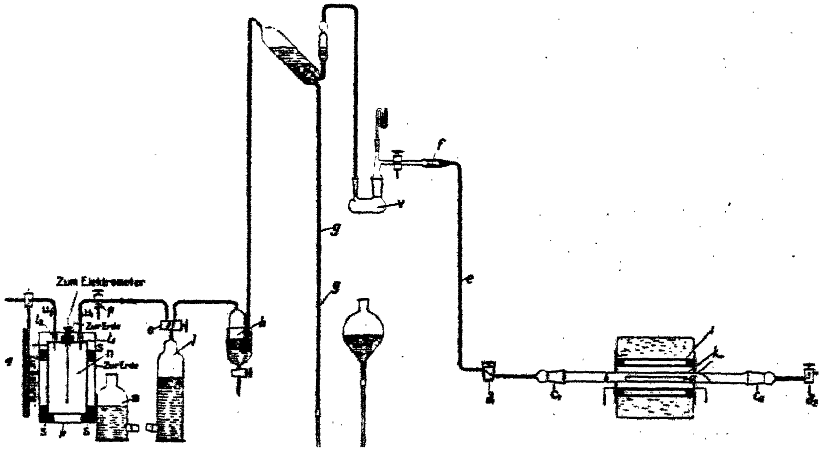
The alcoholic filtrate is evaporated to dryness, the residue moistened with dilute hydrochloric acid and the solution again evaporated. The residue is then taken up in 5 to 10 c.c. of hydrochloric acid (sp. gr. 1·1) transferred to a Rothe separator and iron removed by Rothe's ether process (*Mitt. Kgl. Techn. Versuchs-anstalt*, 1892, 123). The iron is obtained in aqueous solution by shaking the combined ethereal extracts with water, and is determined in any convenient manner.

The acid aqueous layers from the separator are united, warmed to expel dissolved ether and taken down to fuming after addition of 0·5 c.c. of sulphuric acid. The cooled residue is taken up in dilute sulphuric acid, the solution made alkaline with ammonia and any trace of ferric hydroxide filtered off, re-dissolved, and its solution added to the main solution of ferric chloride separated by the ether process. The solution of copper and nickel freed from the last trace of iron is next acidified so that it contains about 5 per cent. of sulphuric acid, and copper is precipitated by passing hydrogen sulphide through the hot (80° C.) solution. The copper is finally obtained as oxide or as copper, and usually carries a weighable trace of nickel which is best determined by the dimethylglyoxime method. The copper may also be accompanied by a trace of platinum, but this never exceeds 0·5 mgrm.

The filtrate from the copper sulphide is evaporated to small bulk, mixed with a saturated solution of ammonium sulphate and 50 c.c. of 25 per cent. ammonia and electrolysed for nickel, preferably using Frary's apparatus (*Zeitsch. Electrochem.*, 1907, 13, 308).

G. C. J.

Estimation of Radium in Minerals and Rocks. E. Ebler. (*Zeitsch. Electrochem.*, 1912, 18, 532-535.)—This paper includes an illustration and fuller description of the apparatus already described by the author (ANALYST, 1912, 323). The parts of the apparatus from the platinum boat *a* to the ground connection *f* can be identified by reference to the abstract of the earlier paper. The drying tube *e* is charged with sodium and not with phosphoric anhydride, which absorbs notable amounts of niton and sometimes gives off lower oxides of phosphorus which make the air a conductor. The use of the pump *g* to transfer the air and emanation to the vessel *h* is obvious from the drawing. Their further transference from *h* to *l* is effected by means of the balance vessel *m*, the tubulure of which is connected to that of *l* by flexible tubing not shown in the drawing. The gas is left in *l* some minutes to allow time for the complete decomposition of any possible thorium emanation and is then transferred to the ionisation chamber *n*, where the measurement of the maximum saturation current is made after four hours. The ionisation chamber carries a manometer, *q*, and rests on the ebonite supports *ss*, inside the metal casing *r*, which is earthed. The ionisation chamber itself consists of a double-walled brass vessel, the space between the walls being filled with tin. Its inner wall is galvanised with zinc.



G. C. J.

Study on the Acidity of Soils. A. Gregoire. (*Bull. Soc. Chim. de Belg.*, 1912, **26**, 336-342, 362-375.)—The estimation of the acidity of a soil is a necessary preliminary for gauging its requirements in lime. The acid constituents of the soil exist largely in a condition in which they are not readily extracted, and the author gives an account of the principal methods which have been proposed for their estimation, and discusses the interpretation of their results in relation to the necessity of liming. He further proposes a method for the estimation of acidity based on the liberation of iodine from a mixture of iodide and iodate as applied by Kjeldahl for the estimation of feeble organic acids. The reagent employed contains 55·3 grms. of potassium iodide, 14·3 grms. of potassium iodate, and 99·2 grms. of crystallised sodium thiosulphate, per litre. The solution of iodine employed for titrating back the excess of thiosulphate contains 17 grms. of iodine and 25 grms. of potassium iodide per litre, and the solutions are standardised against each other by adding 20 c.c. of $\frac{N}{5}$ acid to the first, and titrating back with the iodine. The solution of iodide, iodate, and thiosulphate is called Kjeldahl's solution; it keeps very well. For the estimation, 10 grms. of soil, passed through a sieve with holes of 1 mm., are placed in a stoppered flask with 15 c.c. of the Kjeldahl reagent. After a sufficient time, the volume is made up to 105 c.c., the solution is filtered, and 100 c.c. are drawn off for titrating back the thiosulphate. If the soil be digested with the diluted Kjeldahl solution, the action is very much weaker than with the undiluted reagent. The action of the reagent upon the soil is not instantaneous, but iodine continues to be liberated for a very considerable time, so that it seems difficult to arrive at a final point. The results obtained in several experiments with the same soil are, however, very regular, and it appears desirable to neglect the slow ulterior action of the reagent, possibly on certain mineral constituents of the soil, and to confine the observations to the iodine liberated by the more pronounced organic acids in a period of twenty-four hours. Experiments have shown that the temperature of the digestion within moderate limits has no influence on the results. Further tests have also shown that a large number of inorganic substances behave as acids towards the Kjeldahl reagent; for instance, aluminium sulphate and chloride react with the whole of their acid, and in the case of ferrous ammonium sulphate about half of the sulphuric acid reacts, corresponding to that which is in combination with iron. J. F. B.

Estimation of Total Manganese in Soils. R. A. Gortner and C. O. Rost. (*J. Ind. and Eng. Chem.*, 1912, 4, 522-524.)—As repeated treatment with hydrofluoric acid and sulphuric acid is required in order to obtain in solution all the manganese present in soils, the author recommends the following method as being more rapid. One grm. of the soil is fused with 4 grms. of sodium carbonate, the melt is dissolved in 100 c.c. of water, acidified with 130 c.c. of 35 per cent. sulphuric acid, and diluted to a volume of 250 c.c. Should a precipitate of silica separate at this point it must be removed by filtration, but a slight opalescence does not interfere. One hundred c.c. of the solution are then treated with from 0·25 to 1 grm. of sodium bismuthate, and boiled until the manganese has been oxidised to permanganate. If there is any red coloration the boiling has not been continued long enough. The solution is now cooled, and the coloration compared against a standard permanganate solution which has been prepared by reducing a potassium permanganate solution (in 20 per cent. sulphuric acid) with sulphurous acid and reoxidising the mixture with sodium bismuthate. This standard solution should contain about 0·2 mgrm. of MnO per 10 c.c. Sodium bismuthate is preferred to ammonium persulphate as an oxidising agent as it gives a better oxidation, always giving the true permanganate tint whilst persulphate is liable to produce a reddish tint.

W. P. S.

Quantitative Estimation of Thorium, especially in Monazite Sand, by Means of Sodium Hypophosphate. A. Rosenheim. (*Chem. Zeit.*, 1912, 36, 821.)—The sample (100 grms. in the case of a 5 per cent. monazite sand) is decomposed with concentrated sulphuric acid at 180° to 200° C. in the usual manner, the sulphate dissolved in cold water, and the solution made up to 1,000 c.c. When the undissolved matter has settled, 100 c.c. of the clear solution is pipetted off and mixed with 50 c.c. of concentrated hydrochloric acid and about 180 c.c. of water, and heated to boiling. A cold saturated solution of sodium hypophosphate is then added as long as continued addition gives rise to any further precipitate, and the thorium hypophosphate is filtered off and washed with water acidified with hydrochloric acid until the washings no longer give any precipitate (ceria, etc.) on addition of oxalic acid (or hydrogen peroxide) and ammonia. The precipitate of thorium hypophosphate always contains titanium, and zirconium if present in the sample, but only traces of cerium and other rare earths. Tetravalent cerium gives rise to a very insoluble hypophosphate, but the high acid concentration in which the above precipitation is made ensures the absence of tetravalent cerium. To free the thorium from titanium, zirconium, and traces of other earths, the precipitate and filter-paper are digested with sulphuric acid in a capacious platinum basin or a Kjeldahl flask with occasional addition of a crystal of nitre to hasten the oxidation of the paper. The acid is mostly boiled away, and the cooled mass taken up with water. Since anhydrous thorium sulphate dissolves with difficulty, it economises time to make the solution faintly alkaline with ammonia. When the finely-divided suspension of anhydrous thorium sulphate has given place to a flocculent precipitate of hydroxide, the mixture is made strongly acid with hydrochloric acid, boiled, and the solution of thorium chloride filtered from any trace of silica. In the hot filtrate, thorium is precipitated as oxalate, which is filtered off after twelve hours and washed with dilute hydrochloric acid until free from the last traces of cerium, zirconium, and titanium. The precipitate is finally ignited, and weighed as oxide as usual.

For purposes of works control, useful information may be obtained by igniting the original precipitate of $Th(PO_3)_2$ and weighing it as pyrophosphate, ThP_2O_7. Apart from the fact that the hypophosphate precipitate is never pure, this abbreviated method has another source of inaccuracy in that the oxidation of hypophosphate to pyrophosphate is never quite complete. As a check on manufacturing operations, however, the method is useful.

Sodium hypophosphate is not yet readily procurable, but it can be prepared by the method of Rosenheim and Pinsker (*Ber.*, 1910, **43**, 2003). It will shortly be obtainable in commerce, since a process for working up monazite sand on the large scale by its use has been described. G. C. J.

Analysis of High-Grade Tungsten Alloys. T. Kuczynski. (*Anzeiger Akad. Wiss. Krakau.*, 1911, 542-544; through *Chem. Zentralbl.*, 1912, II., 283.)—When the other constituents of the alloy form readily volatile chlorides, the alloy is conveniently opened up by heating in a current of chlorine. It need not be finely powdered. When the reaction is complete, and the tube in which it is conducted has cooled (in a current of chlorine), this tube and the Peligot tube are washed out successively with dilute hydrochloric acid and dilute ammonia. Excess of hydrochloric acid and about 5 c.c. of nitric acid are added to the liquid, which is heated to boiling, concentrated to about 60 c.c., and then allowed to cool. The separated tungstic acid is filtered off and washed, and the filtrate evaporated to dryness, to throw out of solution the last traces of tungstic acid. The residue is heated to 120° C., to render insoluble the tungstic acid, which is moistened with dilute (1 : 5) hydrochloric acid, filtered off, and washed. The combined tungsten precipitates are redissolved in ammonia, separated from a trace of iron, and the solution evaporated to dryness. The tungstic acid is transferred to a quartz crucible by the use of a little ammonia, excess of nitric acid is added, the liquid evaporated, and the residue ignited and weighed as WO_3. This method is especially useful when a determination of carbon has to be made simultaneously.

If only metals have to be estimated, decomposition of the alloy by means of nitric and hydrofluoric acids is quicker. The fragments of alloy, contained in a platinum dish, are covered with 5 c.c. of nitric acid, and 0·5 grm. of ammonium fluoride is added in small pieces at a time. The reaction, violent at first, needs the heat of a water-bath to sustain it towards the end. To the clear solution 2 or 3 c.c. of sulphuric acid are added, and the solution taken down to fuming. The cooled residue is diluted with water and 20 c.c. of hydrochloric acid, and the tungsten separated as already described. G. C. J.

Measurement of Concentration of Hydrogen Ions in Sea Water. S. Palitzsch. (*Compt.-rend. des Travaux du Lab. de Carlsberg*, 1911, **10**, 85-98.) —The concentration of hydrogen ions in sea water usually lies between $10^{-7\cdot95}$ and $10^{-8\cdot35}$. In a series of measurements made during a cruise from the Baltic to the Black Sea, the only exceptions to this generalisation were certain samples taken from great depths in the Black Sea, which contained hydrogen sulphide, but were still slightly more alkaline than pure water, the highest hydrion concentration noted being $10^{-7\cdot25}$, a number for which a lesser degree of accuracy is claimed than for the others given in the paper.

The method adopted was that of Sörensen (ANALYST, 1909, **34**, 498), in which comparison is made with a series of standard solutions, using an appropriate indicator. For the narrow range of concentrations covered by this inquiry, only two of Sörensen's fundamental standards are needed—namely, $\frac{N}{10}$ hydrochloric acid and a borate solution made by dissolving 0·2 gram-molecules (12·404 grms.) of boric acid in 100 c.c. of $\frac{N}{1}$ sodium hydroxide, free from carbonate, and diluting to 1,000 c.c. with boiled water. These two solutions are then mixed in varying proportions, to obtain a series of substandards. A mixture of 5·25 c.c. of the borate solution with 4·75 c.c. of the hydrochloric acid corresponds to a hydrion concentration of $10^{-7\cdot62}$, whilst 8 c.c. of borate and 2 c.c. of acid correspond to $10^{-8\cdot91}$. The paper includes a table showing the hydrion concentrations corresponding to many intermediate mixtures, together with the corrections to be applied for the influence on the indicator of the high saline concentration of sea water. For hydrion concentrations from $10^{-8\cdot91}$ to $10^{-8\cdot29}$, phenolphthalein is used as indicator; for higher concentrations up to $10^{-7\cdot62}$, a-naphtholphthaleïn is preferred, and is, indeed, necessary, when the concentration exceeds $10^{-8\cdot06}$, since water less alkaline than this is without influence on phenolphthaleïn.

As a general rule the hydrion concentration increases with the depth from which the sea is sampled. A series of samples taken at one spot in the Atlantic showed concentrations varying from $10^{-8\cdot22}$ at the surface to $10^{-7\cdot96}$ at 750 and 1,000 fathoms, and samples collected similarly in other seas yielded very similar results. G. C. J.

Estimation of Zinc in Ferruginous Minerals. E. Beyne. (*Bull. Soc. Chim. de Belg.*, 1912, **26**, 355-361.)—The method generally used for the assay of zinc ores in industrial laboratories is that of Schaffner. The mineral is opened up with nitric acid and hydrochloric acid, the metals of the copper and arsenic group are removed by sulphuretted hydrogen, the filtrate is collected in a 500 c.c. flask, the excess of sulphuretted hydrogen is removed by boiling, and the iron is oxidised by nitric acid. On cooling, 100 c.c. of ammonia (sp. gr. 0·92) are added, and 5 c.c. of hydrogen peroxide if manganese be present. The whole is shaken and allowed to settle until the next day, when it is filtered. Meanwhile a control solution is prepared containing approximately the same amount of pure zinc, the same amount of acid and ammonia, as the solution to be analysed. The titration of the two liquids with sodium sulphide is then conducted simultaneously, until a drop of each produces the same effect upon a strip of lead paper. The principal source of error in this procedure is caused by the co-precipitation of an appreciable amount of zinc with the ferric hydroxide, in spite of the large excess of ammonia employed. The amount of the error increases with the amounts of the two metals present. Various means have been recommended to avoid this error, and the author finds that the simplest consists in adding a comparatively large excess of ammonium chloride before precipitating the iron, a similar quantity of the salt being also added to the control solution. The actual quantity of ammonium chloride necessary has not yet been worked out for minerals containing various proportions of zinc and iron. The extent to which the error is eliminated under given conditions increases with the quantity of ammonium salt added. Thus, with a solution corresponding to 2·5 grms. of a mineral containing 40 per cent. of zinc and 20 per cent. of iron in 250 c.c., an addition of 35 to 40 grms. of ammonium chloride, before precipitating with ammonia and diluting to 500 c.c., gave the theoretical result in the titration for zinc. The addition of more than 40 grms. would begin to interfere with the sharpness of the end-point of the titration.

J. F. B.

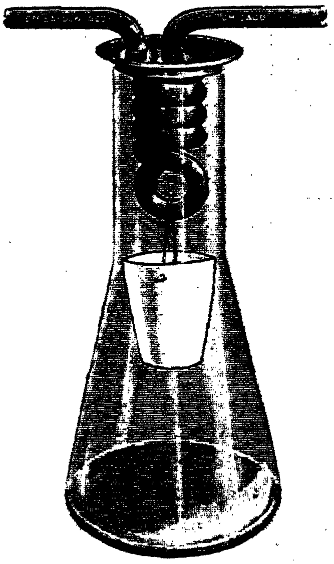
APPARATUS, ETC.

Porcelain Bunsen Burners. **E. Beckmann.** (*Zeit. angew. Chem.*, 1912, **25**, 1515-1518.)—Various forms of Bunsen burners with porcelain tubes are described and recommended for use in spectroscopic work. They may be obtained from Altmann, Luisenstrasse, 47, Berlin.

C. A. M.

Apparatus for the Detection of Carbon Monoxide. **M. A. Guasco.** (*Compt. rend.*, 1912, **155**, 282.)—Two bulbs, one of which is platinised, are carried at the extremities of a vertical U-tube, and are enclosed in a gas-tight envelope, to which the gas to be examined is admitted through a porous septum. The portion of the U-tube outside the envelope contains a coloured liquid, and is so arranged that very small differences of level, produced by differences in temperature of the bulbs due to occlusion of inflammable gases by the platinum, are readily observed. With the apparatus used, the difference in the levels in the two limbs produced by 1 volume of carbon monoxide per 1,000 was 13 mm., and by 2 in 1,000, 26 mm. The smallest quantity giving a distinct indication was 1 in 10,000. If mercury is substituted for the coloured liquid, the apparatus can readily be arranged to close a circuit, and thus release an alarm signal.

O. E. M.



Extraction Apparatus. H. J. Cary-Curr. (*J. Ind. and Eng. Chem.*, 1912, 4, 535-536.)—The apparatus described is for the determination of the constituents of rubber insulation material on wire or cord, and consists of a conical flask 6 inches high, fitted with a metallic spiral tube condenser, passing through a metal plate which forms the cover of the flask. The sample is placed in the suspended Gooch crucible, at the base of which is a disc of filter-paper. The whole is placed on a hot plate or other convenient heater, and works without need of attention, the solvent condensing at the base of the coil and dripping directly into the crucible. The increase in the weight of the conical flask is the weight of matter extracted.

H. F. E. H.

Use of the Zeiss Interferometer in the Technical Analysis of Flue Gases. O. Mohr. (*Zeitsch. angew. Chem.*, 1912, 25, 1313-1317.)—Haber and Löwe (*Zeitsch. angew. Chem.*, 1910, 23, 1393) have described a new interferometer by means of which Rayleigh's principle may be applied to problems of technical gas analysis. They confined themselves, however, to relatively simple problems, such as the determination of methane in mine air and the examination of technical hydrogen. The analysis of flue gases is complicated by the fact that they are not a mere mixture of carbon dioxide with air, nor are they air in which so much oxygen has been replaced by an equivalent amount of carbon dioxide. The sum of the percentages of oxygen and carbon dioxide is always less than the percentage of oxygen in normal air, mainly on account of the hydrogen in the fuel, and there is always the possibility that carbon monoxide may be present. The authors show, however, that the new method of analysis may be extended to flue gases. After being dried by means of sulphuric acid, the gas is passed through the interferometer chamber and a reading taken. It is then passed through a soda-lime tube to deprive it of carbon dioxide, and again through the interferometer, and another reading, which will be lower than the first, is taken. The difference between these first two readings is the measure of the carbon dioxide. With the Zeiss instrument used by the authors a difference of 0·01 scale division corresponds to 0·186 per cent. carbon dioxide. In the absence of carbon monoxide, the second reading also gives the relative proportions of oxygen and nitrogen, which are read off by reference to a straight line curve constructed from readings obtained with mixtures of oxygen and hydrogen in known proportions. From these values for oxygen and nitrogen, which together total 100 per cent., the percentage of oxygen and nitrogen in the original gas is calculated by reference to the percentage of carbon dioxide found. If the percentage of oxygen thus found plus the percentage of carbon dioxide is notably less than it should be, the presence of carbon monoxide is nearly certain. The authors have constructed several tables to facilitate the calculations. The final table is for determining whether carbon monoxide is present or not, and it gives the totals to which the percentages of carbon dioxide and oxygen should sum for four grades of fuel (from coke to brown coal), and with varying excesses of air above that necessary for combustion. G. C. J.

Determination of Melting-Points with the Aid of the Microscope. M. P. Cram. (*J. Amer. Chem. Soc.*, 1912, **34**, 954-956.)—In toxicological investigations the m.pt. of a substance too small in amount to be introduced into a capillary tube may be determined within a few degrees by the use of a compound microscope fitted with a ⅔-inch objective and magnifying 100 diameters. The diaphragm is removed, and beneath the table is clamped a section of brass tube, 3·1 cm. in diameter and 2·4 cm. long, fitted with glass windows at the end, and wound with a heating coil of "nichrome" wire through which is passed an electric current. The heating wire is impregnated with Bakelite varnish No. 2. Four yards of asbestos-covered ·020 nichrome wire are wound on, and the outside wrapped with four layers of asbestos paper. Squares of asbestos board with a hole in the centre are used above and below the tube to prevent loss of heat. The thermometer is introduced through a side-tube, the centre of which is 8 mm. above the bottom of the main tube, and the bulb is entirely within the centre of the coil, the stem extending horizontally to one side. Above the bulb of the thermometer is a wire bridge, upon which the microscope-slide which is to hold the substance is placed. Round cover-glasses 2 cm. in diameter are best adapted to hold the material to be tested. The temperature of the thermometer is not exactly that of the substance on the slide, but the true m.pt. can be found by noting the thermometer reading with substances of known m.pts. A current of 0·6 ampère gave a reading of 64° C., while a temperature of 200° C. was obtained with a current of 1·25 ampères. H. F. E. H.

New Sublimation Apparatus and its Application in Food Analysis. E. Philippe. (*Mitt. Lebensmitteluntersuch. u. Hyg.*, 1912, **3**, 41-53; through *Chem. Zentralbl.*, 1912, II., 82.)—The apparatus consists of a metal capsule of 7 cm. diameter, serving as a condenser, inverted with its lower edge resting on the convex surface of a watch-glass of 11 cm. diameter, with the interposition of a soft rubber ring about 12 mm. wide and 6 to 7 mm. thick, and held in position by three spiral spring clips. Through the top of the capsule pass two tubes of 8 mm. diameter, serving for the circulation of the cooling-water. The watch-glass is placed with its concave side on a flat glass dish with a ground edge, having a height of 3 cm. and a diameter slightly smaller, or at any rate not larger, than that of the capsule. The arrangement rests on a tripod with a double wire gauze. The apparatus is intended for quantitative sublimations, and is applicable for the estimation of salicylic and benzoic acids in foods. Tests made with various sublimable substances showed a yield generally of over 95 per cent. The greatest loss was experienced with vanillin —viz., 5·5 per cent. After each estimation the previously tared watch-glass is wiped on its under side, and the sublimate is dried in the desiccator for half an hour before weighing. In the estimation of salicylic acid in milk an error of about 33 per cent. was found, owing to the fact that some of the salicylic acid was carried down in the precipitation of the milk with copper sulphate and sodium hydroxide; in the estimation of benzoic acid in milk the yield ranged from 84·81 to 96·41 per cent. In fruit preserves and fruit juices the estimation of salicylic acid by sublimation of the ethereal extract gave results agreeing well with the bromine titration method. For the estimation of salicylic acid in a coffee substitute preparation, 10 grms. were freed from fat by petroleum spirit, treated with 10 c.c. of ammonia, extracted with chloroform, and the chloroform extract was submitted to sublimation. The results were in good concordance with the estimation by the Trillich-Göckel method. J. F. B.

Beckmann Thermometer with a Modified Arrangement for setting the Mercury Thread. A. Kuhn. (*Chem. Zeit.*, 1912, **36**, 843.)—The construction of the thermometer is shown in the illustrations. The excess of mercury is brought into the two side limbs by simply inclining the thermometer, and the capillary may be filled by a reverse movement, the lower reservoir of the instrument being at the same time warmed until the mercury thread reached the elongated point *a* of the capillary (Fig. 3). The opening at the point *a* is of such size that each drop of mercury flowing from it measures 1° C. on the scale. It is not necessary to tap the thermometer during these operations. W. P. S.

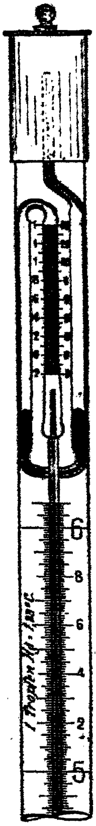


FIG. 1.

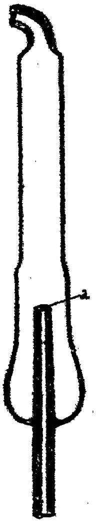


FIG. 3.

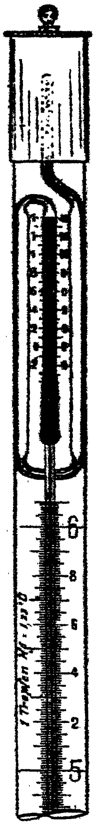
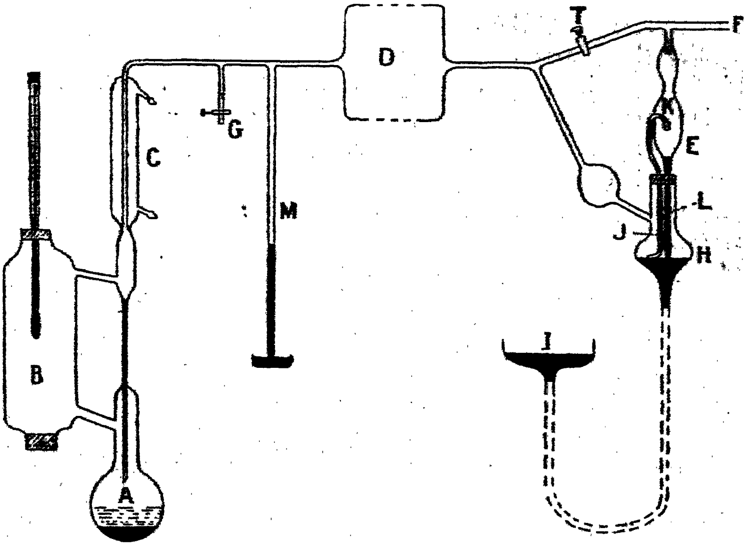


FIG. 2.

New Hand Photometer. W. J. Dibdin. (*J. Soc. Chem. Ind.*, 1912, **31**, 624-625.)—The present instrument is a development of the author's radial photometer described in 1884. It is a portable instrument, weighing 6 pounds, and measuring 17 × 5 × 5 inches, and may be used for comparison of lights in rooms, streets, etc. The instrument is designed to embrace three conditions: (1) Maximum open scale readings; (2) convenience and accuracy in comparing lights of different colours; (3) reliability, by reason of the facility with which its indications can be checked. The open scale readings are obtained by the employment of an electric incandescent lamp, the current to which (from a storage cell) is regulated by means of a rheostat giving an unusually lengthy movement of the contact-point, the position of which is read on a scale graduated in terms of "foot-candles." This arrangement allows of more delicate measurements than by direct readings of the voltmeter, which is also fitted in the instrument to check the working of the storage cell. The comparison of lights of different colours is secured by means of the author's improved form of Leeson's star-disc. Readings of this instrument compared simultaneously with those of a standard photometer show it to give accurate results, the figures differing by +0·01 to −0·04 foot-candles, with an average of −0·01 for twelve readings.

To ascertain the "illuminating power" of any radiant, the square of the distance in feet from the photometer disc is multiplied by the "foot-candle" power observed. As the instrument indicates up to 12 foot-candles, a light at 25 feet distance giving that degree of illumination would have an intensity of 7,500 candles; while if the power at the same distance was only 0·001 candle (lowest observable), the initial intensity would only be 0·625. The range of the instrument is thus wide enough for all ordinary purposes. A. R. T.

Easily Adjustable Vapour Thermostat. J. Fletcher and D. Tyrer. (*Proc. Chem. Soc.*, 1912, **28**, 189-192.)—The authors describe a vapour-bath which can be maintained constant with very little trouble to 0·005° C., and can be fixed at any desired point (say, between 30° C. and 130° C.) with the greatest ease. In principle it consists of a pure liquid boiling under a constant and adjustable pressure, which is controlled and maintained constant by an efficient manostat. Practically any liquid of suitable boiling-point may be used. The boiling liquid is contained in the flask *A* (see diagram). The vapour passes into the compartment *B*, which may be of any particular form suitable; *B* also contains a thermometer, as shown. The vapour is then condensed in *C*, and returns as liquid to the boiling flask *A*. The end of the condenser *C* is connected, as shown, with an air reservoir, *D*, of about 10 to 20 litres capacity (the larger the size of this reservoir the greater is the efficiency of the apparatus), and then to the manostat *E*. The manostat, being the most important part of the apparatus, is described at some length, and for the sake of



clearness has been drawn in the diagram on a rather large scale. The tube *F* is attached to an efficient water-pump. The degree of exhaustion is first roughly adjusted by allowing air to enter by the side-tube and pinch-cock *G*. The mercury rises into the bulb *H* out of the reservoir *I* until the level reaches the end of the tube *J*, when it stops and remains constant. If it passes this point, it closes off the rest of the apparatus from the pump; and as air is entering through the side-tube *G*, the pressure increases, and the level of the mercury in the manostat falls again. As soon as the end of the tube *J* becomes uncovered, the pump again comes into play, and rapidly reduces the pressure, causing the mercury level to rise again. When equilibrium is reached, the level of the mercury remains constant, and a continual current of air and mercury passes up the tube *J* into the compartment *K*, from whence the mercury falls back into the bulb *H* by the tube *L*, and the air passes off through the pump. It will be clear that the difference in the levels of the mercury in *H* and *I* represents the pressure in the apparatus, and the adjustment of this pressure is merely made by altering the height of the reservoir.

For the proper working of the manostat it is necessary to have a large air-supply to draw from, otherwise the mercury level in *H* rises and falls irregularly. This is the purpose of the air reservoir *D*. Although the level of the mercury in *H* may fluctuate slightly, the pressure recorded by the manometer *M* remains perfectly constant. In order to raise the temperature in *B*, the mercury reservoir *I* is raised, and to lower the temperature it is merely necessary to lower *I*.

For the efficient working of the apparatus the author insists that attention should be paid to the following details: The reservoir should be shallow, so that if the vessel *K* partly fills with mercury, as sometimes happens, the difference in the mercury levels in *H* and *I* is not appreciably altered. The end of the tube *J* should be a narrow slit not wider than 0·5 mm., and bent horizontally. The width of this slit insures that the greatest possible variation in the pressure is 0·5 mm., although the actual variation need not exceed 0·1 mm. In order to accelerate the initial exhaustion of the apparatus, the tap *T* is provided as a by-pass, which at all other times must be closed. The connecting tubes should be of wide bore, so that the pressure at all parts of the apparatus is absolutely the same. The boiling liquid used must be pure, and must boil quietly without bumping. It is found that bumping is prevented, however low the pressure, by placing in the flask *A* a layer of mercury as indicated. It is better to boil the liquid over a small free flame shielded from draughts. If the efficiency of the water-pump varies considerably, and if it is desired to have a very constant temperature, it is better to use two manostats connected together with an air reservoir and air inlet tube between them. The temperature can, however, be maintained constant to about 0·01° C. with one manostat.

If rubber stoppers are used, an alcohol should be employed as the boiling liquid, but with ground-glass connections lubricated with glycerol-dextrose mixture any liquid can be used. For temperatures ranging from 30° to 65° C. the authors recommended methyl alcohol, from 40° to 70° C. ethyl alcohol or benzene, and for higher temperatures toluene or xylene.

In experiments made with a Beckmann thermometer in *B*, and with a double manostat, it was found that for short periods of time the temperature remained constant to 0·001° C. For longer periods the variation is greater; but so long as the atmospheric pressure remains unchanged, the temperature remains constant to about 0·005° C.

It is claimed that this form of thermostat has many advantages over the usual large water-bath. It is more quickly adjustable, and need not be left going for long periods of time, as it is only a matter of a few minutes to start it again.

* * * * *

GOVERNMENT REPORTS.

Regulations under the Public Health (Regulations as to Food) Act, 1907.—Sir,—I am directed by the Local Government Board to advert to the Public Health (Regulations as to Food) Act, 1907 (7 Edw. VII., c. 32), under which the Board are empowered to make regulations authorising measures to be taken for the prevention of danger arising to public health from the importation, preparation, storage, and distribution of articles of food or drink (other than drugs or water) intended for sale for human consumption. In connection with the powers thus conferred the Board have for some time had under consideration and inquiry the danger to health which may be entailed by the addition of preservatives and other foreign substances to milk and cream and the importance of controlling such additions, and they have now made Regulations under that Act entitled "The Public Health (Milk and Cream) Regulations, 1912." Copies of an Order containing these Regulations are enclosed.

In making these Regulations due regard has been had to representations which the Board have received from local authorities under the Sale of Food and Drugs Acts, from representatives of the trades affected and from others concerned.

The Regulations are designed to secure that no preservatives shall be added to milk, or to cream containing less than 35 per cent. by weight of milk fat, at any stage from the place of production to that of delivery to the purchaser. In the case of cream containing over 35 per cent. of milk fat, the addition of boric acid, borax, or a mixture of these preservative substances, or of hydrogen peroxide, is not prohibited by the Regulations, but is subject to a system of declaration which is required to be followed by all persons dealing with such cream for the purpose of sale for human consumption. By this system (Part II. of the Regulations) it is intended that preserved cream as an article of commerce shall in all stages be differentiated from cream to which no preservative has been added. Further, Article IV. (*a*) prohibits the addition of any thickening substance to cream or preserved cream.

It will be seen that except as regards Part III. the duty of administering the Regulations has been placed upon those authorities in England and Wales who are Local Authorities under the Sale of Food and Drugs Acts.

Existing arrangements for the collection and examination of milk samples under the Sale of Food and Drugs Acts can readily be extended so as to enable the Council to receive such reports from the public analyst as are requisite. Arrangements will be required for the periodical examination of samples of cream for the purpose of ascertaining whether the requirements of the Order are being duly complied with.

In the case of any contravention of the Regulations, steps should be taken to ascertain the circumstances in which the default has arisen, with a view to any necessary action. Article VI. requires that before the Local Authority institute proceedings against any person for contravention of any of the Regulations in Part II. of the Order, the Local Authority shall afford him an opportunity of furnishing an explanation, and shall consider such explanation and all the circumstances of the case. It should be observed that the proceedings here in question are those authorised by the Public Health (Regulations as to Food) Act, 1907, and the enactments referred to in the Notice at the foot of the Order.

It will be desirable that the Medical Officer of Health should be instructed to exercise general supervision over the action taken in pursuance of these Regulations, and that the officer of the Council who directs sampling under the Sale of Food and Drugs Acts, should be instructed to confer with the Medical Officer of Health and with the Public Analyst as to the details of procedure necessary to secure the observance of the Regulations in the area over which the Council has supervision.

The Regulations will as regards their main provisions take effect on and after October 1 next, and as regards Article V. (1) on January 1, 1913. The Board are anxious to secure uniformity in the administration of the Regulations, and will be glad to have their attention drawn to any cases of difficulty which may hereafter arise.

Copies of the Regulations and of this Circular are enclosed for transmission to the Medical Officer of Health and Public Analyst.

The Regulations and this Circular will be placed on sale, so that copies may shortly be obtained, either directly or through any bookseller, from Messrs. Wyman and Sons, Limited, Fetter Lane, London, E.C.

I am, Sir,
Your obedient Servant,
H. C. MUNRO, *Secretary*.

THE TOWN CLERK *or* THE CLERK TO THE COUNTY COUNCIL.

The draft of the Regulations was published in the ANALYST, 1912, 109-115. In the final form the following alterations have been made:

Page 110 (March issue of the ANALYST), line 7 from bottom, *for* "40" *read* "35."

Page 110, line 4 from bottom, delete "in amount not exceeding 0·1 per cent. by weight."

Page 111, line 13, *for* "June" *read* "October."

Page 111, lines 22 and 24, *for* "40" *read* "35."

Page 111, line 27, delete "in amount not exceeding 0·1 per cent. by weight."

Page 111, delete last 8 lines of page, and substitute:

ARTICLE V.—(1) Every seller of preserved cream shall, in every advertisement, price list, or trade list which is used in connection with the sale of preserved cream for human consumption, describe that article as preserved cream, and in no such document shall any words be contained which might imply that the preserved cream is cream to which a preservative substance has not been added.

Page 112, line 13 from bottom, after "Act" insert "(as amended by subsequent legislation)."

Page 112, line 7 from bottom, after "officer" insert "or to any officer of the Local Government Board."

Page 113, lines 1 and 3, *for* "40" *read* "35."

Page 113, line 6, delete "in amount not exceeding 0·1 per cent. by weight."

Page 114, line 20, delete Labels I. and II. and what follows to line 5 from bottom and substitute:

LABEL I.

PRESERVED CREAM
CONTAINING
BORIC ACID
NOT EXCEEDING
PER CENT.

LABEL II.

PRESERVED CREAM
(PEROXIDE).

3. The size of the declaratory label shall be determined as follows:

Capacity of Receptacle.	Size of Label.
	Inches. Inches.
Not exceeding 1 pint	¾ by 1¾
Exceeding 1 pint	3 by 5

Circular to Local Authorities in England and Wales for the Purposes of the Sale of Food and Drugs Acts, 1875 to 1907.—SIR,—I am directed by the Board of Agriculture and Fisheries to enclose herewith a copy of the Sale of Milk Regulations, 1912.

These Regulations amend the Sale of Milk Regulations, 1901, in so far as they relate to skimmed or separated milk, and replace the limit of 9 per cent. of total milk-solids on which, under Article 3 of those Regulations, a presumption that the milk is not genuine is based, by a limit of 8·7 per cent. of milk-solids other than milk-fat.

Representations have been received from time to time from Local Authorities of districts where there is a trade in skimmed and separated milk that considerable practical difficulties arise in instituting proceedings in cases in which water has been added to skimmed milk, but, owing to the fact that the milk has been imperfectly skimmed, the total solids have not fallen below 9 per cent. Thus a sample containing 1·5 per cent. of fat and 7·5 per cent. of milk-solids other than milk-fat would probably contain over 12 per cent. of added water, but the total solids are up to the limit of 9 per cent. The present Regulations are intended to obviate these difficulties and to facilitate the prevention of the adulteration of skimmed milk with water.

The fixing of the percentage at 8·7 may result in some hardship in cases where the milk is imperfectly skimmed. Thus, if milk containing 3 per cent. of milk-fat and 8·5 per cent. of milk-solids other than milk-fat is imperfectly skimmed, so that 1·5 per cent. of milk-fat remains, the proportion of milk-solids other than milk-fat will rise only to 8·63 per cent., and the Board would suggest that this point should be taken into account by your Local Authority in considering the question whether they should institute proceedings on the presumption of adulteration based on the Regulations in cases where the deficiency of milk-solids other than milk-fat is slight and the skimmed milk contains a considerable proportion of milk-fat.

I am to enclose duplicate copies of this Circular and of the Regulations for transmission to the Public Analyst, and to say that the Board will be glad to supply further copies of the Circular for distribution amongst the members of your Local Authority and the officers concerned in the administration of the Acts on being informed of the number you require.

I am, Sir,
Your obedient Servant,
T. H. ELLIOTT, *Secretary.*

THE CLERK TO THE LOCAL AUTHORITY.

BOARD OF AGRICULTURE AND FISHERIES,
August 20, 1912.

Statutory Rules and Orders, 1912, No. 687: Adulteration (Sale of Food and Drugs Acts)—The Sale of Milk Regulations, 1912. *(Dated June* 29, 1912.)—The Board of Agriculture and Fisheries, in exercise of the powers conferred on them by Section 4 of the Sale of Food and Drugs Act, 1899, do hereby make the following Regulations:

Skimmed or Separated Milk.—(1) Where a sample of skimmed or separated milk (not being condensed milk) contains less than 8·7 per cent. of milk-solids other than milk-fat, it shall be presumed for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907, until the contrary is proved, that the milk is not genuine, by reason of either the addition thereto of water or the abstraction therefrom of milk-solids other than milk-fat.

Revocation of Existing Regulation.—(2) Regulation 3 of the Sale of Milk Regulations, 1901, is hereby revoked, but not so as to affect the validity of any certificate of analysis given before the commencement of these Regulations or any legal proceedings in respect of any sample to which any such certificate relates.

Commencement.—(3) These Regulations shall come into operation on the First day of September, One thousand nine hundred and twelve.

Extent.—(4) These Regulations shall extend to England and Wales.

Short Title.—These Regulations may be cited as the Sale of Milk Regulations, 1912.

In witness whereof the Board of Agriculture and Fisheries have hereunto set their official seal this twenty-ninth day of June, One thousand nine hundred and twelve.

(L.S.) T. H. ELLIOTT, *Secretary*.

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REVIEWS.

PRINCIPES THÉORIQUES ET PRATIQUES D'ANALYSE MINÉRALE. G. CHESNEAU. Ch. Béranger, Paris. 1912. Price 25 francs.

The author describes this book as a guide for engineers to the analysis of industrial minerals, but it is more truly a comprehensive treatise on inorganic analysis; and though written from the standpoint of the works manager or the mining engineer, it will be of considerable value to the professional chemist.

The first part, which deals with theoretical principles and general methods of analysis, is exceptionally good. It sets forth the *rationale* of analytical manipulation with a completeness and lucidity all too rare in professedly "practical" handbooks. The theory of washing precipitates is fully discussed, and the relation of solubility to size of particles is considered, and the reason why digestion usually diminishes the fineness of a precipitate is indicated. Gelatinous precipitates and the precautions necessary in dealing with them receive full consideration. The equilibrium conditions of the reactions suitable for analytical purposes are discussed with a view to the elucidation of the two fundamental problems—to produce a precipitate as little soluble as possible, and to dissolve insoluble bodies.

Qualitative analysis is next considered; general methods and a complete scheme for the detection of metals are given.

The first part closes with an excellent chapter on the general processes of quantitative analysis. The degree of accuracy attainable in volumetric processes is discussed, and emphasis is laid upon the advantages of the comparative method, in which a "standard" is treated in exactly the same way as the sample.

In the second part the elements are studied separately. The principal sources and uses are first given for each element and its industrially valuable compounds; then its distinguishing characteristics and the analytical properties made use of for its detection and estimation are described. This is followed by an account of selected methods for the separation and estimation of the element in question, and the application of these methods to minerals and manufactured products.

On the whole, the methods chosen are authoritative and reliable, though some are rather unfamiliar to English practice. All are very carefully described, with due attention to such points as concentration of solutions, temperature, strength of acid, etc. Following the treatment of hydrogen, the general question of acidity is dealt with and the theory of indicators outlined. A section is also devoted to water analysis. Phosphorus and phosphoric acid are well treated. Under "Arsenic" the method of Fresenius and Babo is given. This allows of the detection of one-thousandth of a milligram of arsenious acid, and its differentiation from antimony. The consideration of carbon is followed by methods for the estimation of common hydrocarbons, elementary organic analysis, and the analysis of fuels. The chapter on the iron group opens with a clear discussion of the conditions necessary for the separation of the metals as sulphides and acetates. Somewhat surprising is the absence of any mention of the bichromate method for iron estimation.

The analysis of iron and steel is treated in considerable detail. Besides the usual dry and wet combustion methods for carbon, a volumetric method is described. The apparatus appears to be rather complicated, and, as only one determination can be made in two hours, its advantages are not very obvious. The Eggertz colour method is reviewed, with due warning as to its limitations. For sulphur, the only oxidation process described is the "Arnold" method, in which aqua regia is used together with bromine and potassium chlorate, and the precipitation with barium chloride is made in the cold. The molybdate method described for phosphorus, involving two precipitations of the phosphomolybdate, is decidedly tedious and complicated. No magnesia method is given. Several colour methods for manganese are described, including the persulphate process, which is certainly the simplest and most expeditious. In dealing with the "rare" elements, a preliminary treatment by the ether method of Rothe is recommended. This accomplishes a nearly complete separation of the iron from all the other elements except molybdenum. The estimation of nickel by means of dimethylglyoxime is described, but the cyanide titration in presence of ammonium citrate is not mentioned.

The metals of the copper and tin groups are well treated, and methods for the analysis of their alloys are given.

Like most French publications, the book lacks an index, but there is an alphabetical list of minerals of which the analysis is given.

The printing is good, and the diagrams are clear and well chosen.

F. W. HARBORD.

THE ANALYST'S LABORATORY COMPANION. Fourth Edition. By ALFRED E. JOHNSON, B.Sc. (Lond.), F.I.C., A.R.C.Sc.I. 1912. London: J. and A. Churchill. Pp. ix + 164. Interleaved with blank pages.

As a small work of reference for chemical and physical data, this edition of Mr. Johnson's book deserves commendation. The numerous tables have been revised where necessary, and some further tables have been added, the International Atomic Weights for 1912 being adopted. Considering, however, that refractometric methods of analysis are being employed to an increasing extent, it is rather surprising that space has not been found for tables showing the relation between refractive index and percentage content of solutions of various substances.

The notes on analysis have also been revised and amplified, some very useful additions being the standards for sewage effluents recommended by the Royal Commission on Sewage Disposal (Fifth Report, issued in 1908), and the chief provisions of the Draft of "The Public Health (Milk and Cream) Regulations, 1912," issued by the Local Government Board. In the section dealing with the cupric reducing power of the carbohydrates, a method for the gravimetric estimation of reducing sugars is described, but no mention is made of the volumetric method now so generally used.

Considerable space is devoted to chemical calculations. At p. 36, Mr. Johnson justly condemns the calculation of analytical results to an unwarrantable number of decimal places, but falls into the same error himself at p. 107, where the percentages of dextrose and maltose in a commercial "glucose" are given to the second decimal place.

On the whole, however, the revision has been done very carefully, the errors observed are few, and for the most part trivial, and the new material has been judiciously selected. The book is of a convenient size, and should prove very useful in the laboratory. LEWIS EYNON.

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INSTITUTE OF CHEMISTRY.

PASS LIST: JULY EXAMINATIONS, 1912.

Of thirty-five candidates who presented themselves for the Intermediate Examination, eighteen passed: C. A. Adams, B.Sc. (Lond.), Miss D. J. Bartlett, L. O. Brekke, B.Sc. (Leeds), H. B. Brown, F. G. Conyers, E. D. Goddard, B.Sc. (Lond.), J. B. Gray, F. A. Hatch, M. Howie, D. W. Kent-Jones, E. Marsden, Miss E. G. Mocatta, B.Sc. (Lond.), E. A. Rayner, B.Sc. (Lond.), W. S. Ritchie, B.Sc. (Lond.), W. G. Saunders, D. E. Sharp, B.Sc. (Aberdeen), T. F. Smeaton, and W. A. Storey. Of thirty-seven candidates who presented themselves for the Final Examination, twenty-eight passed. In the branch of Mineral Chemistry: C. R. Chown, A.R.C.S. (Lond.), B.Sc. (Lond.), H. Gilmour, W. P. Harmsworth, J. G. King, H. Knight, P. G. Knapman, B.Sc. (Lond.), and R. H. Truelove, B.Sc. (Lond.), A.R.C.S. (Lond.). In the branch of Metallurgical Chemistry: E. O. Jones, B.Sc. (Lond.), A.R.C.S. (Lond.), W. A. C. Newman, B.Sc. (Lond.), A.R.C.S. (Lond.), and E. W. Yeoman, B.Sc. (Lond.), A.R.C.S. (Lond.). In the branch of Physical Chemistry: J. I. Crabtree, B.Sc. (Vict.), and Miss A. M. Finney, B.Sc. (Birm.). In the branch of Organic Chemistry: J. L. S. Allan, J. Crawford, G. J. Eastburn, J. H. Naylor, M.Sc. (Manc.), F. G. Rawling, M.Sc. (Leeds), S. Robertson, A. Wilson, T. A. Wilson, and T. Wright. In in the branch of the Chemistry of Food and Drugs, and of Water: S. W. Bunker, B.Sc. (Lond.), D. H. B. Cowman, B.Sc. (Lond.), A. Gemmell, B.Sc. (Edin.), C. L. Hinton, H. Raistrick, B.Sc. (Leeds), M. J. Robb, B.Sc. (Aberdeen), and S. B. Tallantyre.

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OCTOBER, 1912 Vol XXXVII., No 439.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE IODIMETRIC TITRATION OF SULPHITES IN PRESENCE OF ALCOHOL AND SUGARS.

By JULIAN L. BAKER, F.I.C., and F. E. DAY, B.Sc., A.I.C.

In a paper published recently (*J. Inst. Brewing*, 1911, **17**, 465; Analyst, 1911, **36**, 495), dealing with the behaviour of sulphites added to beer for preservative purposes, we showed that the bulk of the sulphite combines with various constituents of the beer, with the formation of stable, chemically inert bodies. We based this conclusion partly on the differences between the results obtained by direct titration and by the distillation method. K. Farnsteiner (*Zeitsch. Untersuch. Nahr. Genussm.*, 1902, 5, 1124) employed a method for estimating sulphites in light-coloured liquids, such as lime-juice, which consisted in adding alkali and then acid previously to titration. We found that under these conditions a higher value for sulphurous acid is obtained than by direct titration.

Among several constituents of beer, whose action on sulphites we tested separately, was alcohol, industrial spirit, partially purified by repeated distillation, being employed. We have considered it desirable to repeat our experiments, using pure alcohol; and, contrary to our expectations, we have found that the results obtained by Farnsteiner's method were considerably lower than by direct titration.

Potassium hydrogen sulphite was made up to $\frac{N}{20}$ strength with (*a*) distilled water containing 5 per cent. by volume of pure alcohol, and (*b*) distilled water alone. Twenty c.c. portions diluted with water were titrated with $\frac{N}{10}$ iodine under varying conditions:

Time after Preparation of Solution	C c. Iodine used.		
	Direct Titration.	After Acidifying with HCl.	Farnsteiner's Method.
5 per cent. alcohol:			
½ hour	9·70	9·2	8·90
20 hours	9·55	—	9·10
70 ,, ...	9·40	9·0	8·95
No alcohol:			
At once	9·80	9·3	8·80
70 hours	9·40	—	8·65

Though the results are unaffected by the presence of alcohol, which plainly does not combine with the sulphite, yet the difference in the values of the second and third columns as compared with the first is marked, and in the opposite sense to that anticipated. The purity of the reagents was beyond question, and, if the acid and alkali were previously mixed and then added to the sulphite solution, a normal titration result was obtained. It seemed probable to us that the oxygen dissolved in the liquid might be the cause of this difference, and the following experiments were made to throw some light on the matter: A $\frac{N}{10}$ solution of potassium hydrogen sulphite was titrated with $\frac{N}{10}$ iodine under varying conditions, 70 c.c. of either distilled or tap water (both saturated with air, unless otherwise stated) being used to dilute the liquid before titration. In the table, the results obtained by diluting with distilled and tap water are recorded in separate columns, and the constituents of the mixture titrated are given in the order in which they were added. The total volume titrated in most cases was 80 c.c., which contains approximately 0·0007 grm. dissolved oxygen:

Mixture in Titration Flask.	Time between Mixing and Titrating.	C.c. Iodine used.	
		70 c.c. Distilled Water.	70 c.c.* Tap Water.
5 c.c. sulphite + 70 c.c. water	At once	5·0	4·9
5 c.c. sulphite + 2 c.c. concentrated HCl	At once	5·0	4·6
	7 minutes	—	4·6
5 c.c. sulphite + 70 c.c. water + 5 c.c. 6 per cent. NaOH + 2 c.c. HCl	At once	4·3	4·8
	3 minutes	—	4·3
5 c.c. sulphite + 70 c.c. freshly-boiled water and 5 c.c. 6 per cent. NaOH + 2 c.c. HCl	At once	4·8	—
5 c.c. sulphite + 70 c.c. freshly-boiled water + 5 c.c. concentrated HCl	At once	4·8	—
70 c.c. water + 5 c.c. 6 per cent. NaOH + 2 c.c. HCl + 5 c.c. sulphite	At once	5·0	5·0
	7 minutes	—	4·7

It is evident that in dilute acid solutions the sulphite rapidly combines with the dissolved oxygen, and it seems likely that the action is hastened by the act of neutralising the added alkali, thus exhibiting the phenomenon of an "induced reaction." The amount of oxygen dissolved in the solution is capable of oxidising 0·9 c.c. of $\frac{N}{10}$ sulphite. It will be noted that the addition of a considerable quantity of hydrochloric acid appreciably lowers the titration value. The experiments plainly show the inaccuracy of estimating the amount of sulphurous acid in liquids by Farnsteiner's method.

Having traced the source of the discrepancy in the previous results to the disturbing effect of dissolved oxygen, and having also shown that pure alcohol does not combine with sulphites, experiments similar to those described in our previous paper were carried out with alcohol derived from various sources. A $\frac{N}{10}$ solution of potassium hydrogen sulphite was prepared, containing 5 per cent. of the alcohol under examination, and titrated with iodine. In the table, the column headed "real loss" represents the difference between the amount of sulphurous acid actually present after four weeks, as determined by the distillation method, and that originally present. The "apparent loss" is the difference between the amount shown by direct titration and that originally present; this value includes the "real loss," together with the amount of sulphite in stable combination. The difference between the "apparent loss" and the "real loss" is a measure of the sulphite in combination:

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* This water had a distinct alkaline reaction to methyl orange.

	C.c. $\frac{N}{10}$ Iodine Solution used.						Expressed as c.c. $\frac{N}{10}$ Iodine.			
	By Direct Titration after—				After 4 Weeks.		Apparent Loss.		Real Loss.	Combined SO_2.
	½ Hour.	4 Hours.	23 Hours.	50 Hours.	Direct.	Distilled.	23 Hours.	4 Weeks.	4 Weeks.	4 Weeks.
Pure alcohol ...	10·00	9·90	9·90	9·9	9·20	9·40	0·10	0·80	0·60	0·20
Alcohol distilled from old ale ...	9·70	9·95	9·95	9·8	9·35	9·55	0·05	0·65	0·45	0·20
Methylated spirit ...	9·45	9·45	9·25	9·2	9·10	9·60	0·75	0·90	0·40	0·50
Brandy	10·15	9·90	9·90	9·8	9·70	9·90	0·10 0·25	0·30 0·45	0·10 0·25 *	0·20

It would be expected that the "real loss" would be greater where least combination had occurred, but this idea is hardly borne out by the last column. For example, the "real loss" is far less in the case of brandy than in that of pure alcohol, whereas, according to the results, the amount of sulphite in combination is the same. As regards odour and flavour of sulphurous acid, the test solutions were arranged as above, most odour—and it is to be supposed least combination—occurring in the solution with pure alcohol, and least odour and most combination in the brandy solution. This was confirmed by blowing a litre of air, during a seven and a half minutes' period, through 10 c.c. of each. On titration with iodine the following figures were obtained:

	Pure Alcohol.	Alcohol from Old Ale.	Methylated Spirit.	Brandy.
Before	9·20	9·35	9·10	9·70
After	8·75	9·10	9·05	9·60
Loss	0·45	0·25	0·05	0·10

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* The diluted brandy showed a slight initial absorption of iodine, which has been allowed for in the lower set of figures.

These figures confirm the previous "real losses," and from these experiments we infer that at least two states of combination exist between sulphites and some constituent of alcohols of commercial origin. One results in either decreased volatility of the sulphurous anhydride, or resistance to oxidation by molecular oxygen; the other, more stable, resists even oxidation by iodine. The latter state seems to occur but slightly in the case of pure alcohol, or alcohol derived from fermented liquids, but is exhibited in a marked degree even by good quality methylated spirit.

In our previous paper we showed that there was a considerable amount of combination between maltose and dextrin and sulphites. We have extended these observations by including glucose and cane-sugar. A 10 per cent. solution of the sugar was mixed with $\frac{N}{5}$ potassium hydrogen sulphite and titrated with $\frac{N}{10}$ iodine:

Sugar.	C.c. Sugar Solution.	C.c. $\frac{N}{5}$ $KHSO_3$.	Duration before Titration.	C.c. $\frac{N}{10}$ Iodine.
Glucose	5·0	10·0	—	19·4
"	10·0	10·0	10 minutes	19·4
"	50·0	10·0	10 minutes	19·3
Sucrose	10·0	10·0	—	20·0
"	40·0	10·0	—	20·0
"	50·0	10·0	5 minutes	20·0

The apparent loss of sulphite caused by the addition of glucose is striking, and can probably be correlated with its aldehydic constitution, which would explain why cane-sugar does not show any such effect.

The conclusions to be derived from the preceding experiments are, we consider, as follows:

1. That sulphurous acid is readily oxidised by dissolved oxygen in dilute acid solution.

2. That this action is greatly facilitated by the occurrence in the solution of a reaction between alkali and acid. This obviously affects the results obtained by the method of sulphite estimation proposed by Farnsteiner (*loc. cit.*).

3. That commercial alcohols and alcoholic liquids contain substances capable of combining with sulphites. These substances partially prevent the oxidation of sulphites by iodine.

4. That glucose, by virtue of its aldehydic structure, combines to a certain extent with sulphites, but cane-sugar does not.

STAG BREWERY, LONDON.

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THE GRAVIMETRIC SEPARATION OF ZINC AND NICKEL.

BY T. COCKBURN, F.I.C., A. D. GARDINER, AND J. W. BLACK.

THE unsatisfactory results obtained by the commoner methods for the separation of zinc and nickel renders the analysis of German silver a matter of some difficulty.

For the purpose of our inquiry we selected a few of the better-known processes, omitting those involving the use of cyanides or fixed organic acids, whilst we directed special attention to obtaining zinc sulphide in a form in which it could be filtered—a desideratum which is frequently unattainable.

In order to obtain strictly comparable results, the same standard solutions of zinc and nickel and the same graduated glass apparatus were used throughout.

1. *Precipitation of Zinc as Zinc Sulphide from an Acetic Acid Solution.*—We found considerable difficulty in finding the correct conditions when employing this process. In many of our trials, either the precipitation of the zinc or the physical condition of the zinc sulphide proved unsatisfactory, but by adhering to the following details our best results were obtained:

To a faintly acid solution of the two metals a solution of sodium carbonate is added until a faint turbidity is produced, then 100 c.c. glacial acetic acid, the solution thoroughly cooled, and hydrogen sulphide passed into it for one hour at the rate of about 2 bubbles per second (*cf.* Lewis, *J. Soc. Chem. Ind.*, 1909, **78**, 587).

These conditions insure the production of zinc sulphide perfectly white in colour, and in a granular form permitting of easy filtration. The precipitated zinc sulphide is allowed to stand in the cold for some hours, and the clear supernatant liquid then completely decanted through a hardened Schleicher and Schüll No. 5 paper. The precipitate is washed with sulphuretted hydrogen water containing 2 per cent. ammonium acetate. After drying, the precipitate is separated from the paper, and ignition started at a low red heat, the temperature being gradually increased until a bright red heat is obtained. The duration of the ignition at a gradually rising temperature lasted one hour, when the zinc sulphide was entirely converted into zinc oxide.

The precipitates were tested for nickel by the exceedingly delicate reaction discovered by Tschugaeff (*Chem. Zeit. Rep.*, 1905, **29**, 247)—namely, the production of a dark scarlet nickel glyoxime when to the dilute hydrochloric acid solution of the precipitates, made slightly ammoniacal, was added an alcoholic solution of dimethylglyoxime:

TABLE I.

No. of Experiment.	Weight of Zinc taken.	Weight of Nickel taken.	No. of c.c. of Glacial Acetic Acid.	Total Volume of Solution.	Weight of Zinc found.	Difference.
1	0·1102	0·1934	40	210	0·1105	+0·0003
2	0·2203	0·1934	50	260	0·2211	+0·0008
3	0·3672	0·1934	100	260	0·3675	+0·0003
4	0·3672	0·1934	100	260	0·3668	−0·0004
5	0·6793	0·1934	100	260	0·6791	−0·0002

The results were negative in the instances shown in Table I., but we found that when using larger quantities of nickel there was a tendency for nickel to appear in the zinc precipitate, and consequently high results were obtained.

2. *Precipitation of Zinc as Zinc Sulphide in Presence of Formic Acid (Waring's Method).*—This process, which we prefer to the acetic acid method, was operated as described in Low's "Technical Methods of Ore Analysis," 5th ed., p. 294.

To the acid solution of zinc and nickel is added a drop of methyl orange, then from a pipette a very dilute solution of sodium hydroxide is run in until the pink colour barely but permanently changes to a light yellowish tint. Then sufficient formic acid (50 per cent. strength) is added drop by drop just to restore the permanent pink colour, and ½ c.c. is added in addition. The solution is diluted to about 250 c.c. (or so that 100 c.c. contains not more than 0·2 grm. of metallic zinc), and heated to about 80° C. Sulphuretted hydrogen is then passed through the solution under a slight pressure, and when the zinc has been completely precipitated the flask is disconnected, the precipitate allowed to subside, and the clear supernatant liquid passed through a hardened No. 5 filter or ordinary Swedish filter-paper, and the precipitate washed with hot water. As in the foregoing process, the zinc was weighed as zinc oxide:

TABLE II.

No. of Experiment.	Weight of Zinc taken.	Weight of Nickel taken.	Weight of Zinc found.	Difference.
	Grm.	Grm.	Grm.	
1	0·3672	0·0704	0·3684	+0·0012
2	0·1836	0·0704	0·1844	+0·0008
3	0·1836	0·1760	0·1827	−0·0009
4	0·0734	0·1760	0·0724	−0·0010
5	0·0734	0·3521	0·0730	−0·0004
6	0·2570	0·3521	0·2580	+0·0010
7	0·0734	0·7042	0·0769	+0·0035

The precipitates were tested for nickel by dimethylglyoxime. Positive results were obtained in Nos. 6 and 7. We found this method rapid and easily worked, whilst the separation, except in the case of No. 7, which contained a high proportion of nickel, was clean and sharp.

3. *Precipitation of Zinc as Zinc Sulphide from a Solution faintly Acidified with Hydrochloric Acid.*—The results obtained by this method were unsatisfactory, and after an extended trial it was abandoned. As we anticipated, the main difficulty was to determine the quantity of free acid to employ, whilst the constantly increasing proportion of highly ionised free acid militates against accurate results being obtained.

4. *Zimmermann's Process* (*Ann. d. Chem. und Pharm.*, 1879, **199**, 3; 1880, **204**, 226).—More was expected of this method, but notwithstanding the fact that we carried out the process exactly as described in Treadwell, vol. ii., p. 127, we were unable to obtain concordant results.

With a view to estimating the nickel in the filtrates from the above, we reviewed the recent work of L. Dede (ANALYST, 1911, **36**, 598), which promised to obviate the error arising from the well-known occlusion of alkali by nickel hydroxide. Dede employed potassium persulphate instead of bromine as the oxidising agent, and his results indicated an accuracy of ±0·2 mgrm. on weighings up to 0·3 grm.

The results obtained by us did not conform to this degree of accuracy, notwithstanding the fact that the amount of alkali required in each test was calculated, and great care taken to employ only the smallest possible excess. We found that the precipitates and the nickel reduced therefrom invariably contained alkali, the amount of occluded matter being approximately proportional to the concentration of the nickel. The following results, which are typical of many obtained, plainly indicate this:

TABLE III.

No. of Experiment.	Nickel taken.	Nickel found.	Difference.
	Grm.	Grm.	Grm.
	0·0704	0·0747	+0·0043
	0·1408	0·1516	+0·0108
	0·2464	0·2598	+0·0134

Separation of Nickel from Zinc.—In close connection with the foregoing work, we made an examination of the dimethylglyoxime method of separating nickel from zinc as detailed by O. Brunck (ANALYST, 1907, **32**, 431). To the solution, first neutralised with dilute ammonia and rendered faintly acid with dilute hydrochloric acid, is added a 1 per cent. alcoholic solution of dimethylglyoxime in amount equivalent to seven times the quantity of nickel present. This was followed by the addition of a few c.c. of 10 per cent. sodium acetate solution. The solutions were maintained at the boiling-point for half an hour, filtered through a Gooch crucible having an asbestos felt, and washed with hot water. The precipitate was weighed as $Ni(C_4H_7O_2N_2)_2$, after drying at 110° C.

TABLE IV.

No. of Experiment.	Weight of Nickel taken.	Weight of Zinc taken.	Weight of Nickel found.	Difference.
	Grm.	Grm.	Grm.	Grm.
1	0·0704	—	0·0714	+0·0010
2	0·0704	0·0734	0·0709	+0·0005
3	0·0704	0·3672	0·0712	+0·0008
4	0·0704	0·5508	0·0724	+0·0020
5	0·1408	—	0·1407	−0·0001
6	0·1408	0·1468	0·1406	[illegible]
7	0·1408	0·3672	0·1410	[illegible]

The nickel glyoxime forms an extremely bulky precipitate, and we found that the concentration of 0·07 grm. of nickel per 250 c.c. was as high as desirable.

The process is applicable to the determination of nickel in the filtrates obtained from the separation of zinc after the removal of the sulphuretted hydrogen, formic acid, etc. The results obtained are shown in Table IV.

One of us is making an examination of the dicyan-diamidine method for the estimation of nickel with special reference to German silver, and will shortly publish his results.

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* * * * *

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

ANALYSIS OF FOOD AND DRUGS.

Estimation of Benzaldehyde in Maraschino Cherries and Liqueur. A. G. Woodman and L. Davis. (*J. Ind. and Eng. Chem.*, 1912, 4, 588-589.)—The following method is recommended for this estimation, which affords a means of determining the genuine nature of these products: 100 c.c. of the liquor from Maraschino cherries, or 50 c.c. of the liqueur, are diluted with water to 140 c.c., and 110 c.c. distilled over. The amount of alcohol in the distillate is estimated approximately from the sp. gr., and sufficient alcohol or water added to 100 c.c. of the liquid to make its content of alcohol approximate to 10 per cent. by volume. One hundred c.c. of the phenylhydrazine reagent (freshly prepared by thoroughly shaking 3 c.c. of glacial acetic acid with 40 c.c. of water and 2 c.c. of pure phenylhydrazine, and filtering till clear) is added as soon as possible after the distillate is obtained, and the mixture vigorously shaken for ten minutes. The liquid is filtered through a tared Gooch crucible, washing the precipitated hydrazone with cold water, and finally with 10 c.c. of dilute alcohol (10 per cent.). A rotary motion of the liquid during filtration prevents loss of hydrazone, which has a tendency to adhere to the sides of the vessel. The precipitate is dried in a vacuum desiccator for twenty-four hours, or at 70° to 80° C. in a vacuum oven for three hours, keeping the precipitate away from strong light. A blank experiment must be made, and the weight of precipitate deducted. The weight of hydrazone corrected, multiplied by 0·5411, gives the weight of benzaldehyde present. The method is applicable to the determination of the benzaldehyde in almond extracts, using 10 c.c. of the extract diluted to 100 c.c. with water, and 15 c.c. of the reagent. Control experiments with pure benzaldehyde showed the method to give results from 95 to about 98 per cent. of the theoretical. Commercial samples of Maraschino cherries were found to contain from 4 to 36 mgrms. of benzaldehyde per 100 c.c., and the liqueurs from 0 to 41 mgrms. per 100 c.c. Genuine Maraschino appears to have a very low benzaldehyde content, and Maraschino cherries showing a relatively large amount of the aldehyde are in all probability artificially flavoured. A. R. T.

Estimation of Crude Fibre. E. Gury. (*Mitt. Lebensmitteluntersuch. u. Hyg.*, 1912, 3, 99-105; through *Chem. Zentralbl.*, 1912, II., 556-557.)—The author has submitted filter-paper, cocoa, cocoa-shell, and sawdust to König's process (*Unters. landw. u. gew. Wichtiger Stoffe*, third edition, p. 249), Parry's modification (*Chemist and Druggist*, 1911, 167) of Henneberg's process, Filsinger's process, and to a modification, now described, of the Zeisel-Stritar process (*Ber.*, 1902, 35, 1252). The modification consists in treating the finely-divided, fat-free substance (1 to 1·5 grm.) with 200 c.c. of water and 10 c.c. of concentrated lactic acid, the whole being boiled under a reflux condenser for thirty minutes. The function of the lactic acid is to render the material more open to the attack of the reagents used subsequently; it also shortens the time required for filtration. The insoluble matter is filtered off on an ordinary filter-paper, washed, and returned to the flask with 180 c.c. of water and 20 c.c. of nitric acid, and, after digesting for thirty minutes, is submitted to the process of Zeisel and Stritar.

König's method, as well as that of Parry, leads to the hydrolysis of from 13 to 16 per cent. of the cellulose in filter-paper, whereas the method of Zeisel and Stritar does not attack cellulose. Yet the methods of König and Parry frequently discover more than 50 per cent. more crude fibre in cocoa or cocoa-shell than is found by the author's modification of Zeisel and Stritar's method. This is due to the fact that, even with very fine division of the sample, the interior portions of the larger particles escape the action of the reagents, unless opened to this action in some such way as that now suggested. Even when using his lactic acid method, the author subjects cocoa and cocoa-shell to special methods to secure fine division of the sample. The paper includes a description of a small winnowing-machine of simple construction. By means of this machine the finer particles of a small sample are driven forward, and the heavier residue is repeatedly ground and returned to the machine until all is fine enough.

In view of the small difference in crude fibre content of cocoa mass (5·5 to 8 per cent.) and cocoa-shell (11 to 18 per cent.), the author thinks that an adverse judgment on cocoa should never be based on the percentage of crude fibre alone. On the other hand, he thinks the Swiss *Lebensmittelbuch* is unduly lenient in fixing 11 per cent. as the permissible limit of crude fibre in cocoa. Though König's method may show nearly 9 per cent. of crude fibre in some genuine samples, the author's process never discovers as much as 8 per cent. Filsinger's method is much too crude to give results of any value in examining cocoa for shell. G. C. J.

Modification of the Sweeney Method for Crude Fibre. C. Kennedy. (*J. Ind. and Eng. Chem.*, 1912, 4, 600-601.)—Sweeney (*U.S. Dept. Agri. Bur. Chem. Bull.*, No. 137, 157) has modified the U.S.A. official method of fibre determination by dispensing with the filtration of the material after boiling with acid. He merely neutralises the whole with 10 per cent. caustic soda and then adds 200 c.c. of boiling 2·65 per cent. caustic soda for the final boil. This neutralisation, the author [illegible] out, throws down a certain amount of the material dissolved by the acid, and thereby introduces an error, since the precipitate will be weighed as fibre. It is recommended that, instead of neutralisation as carried out by Sweeney after boiling with acid, sufficient boiling caustic soda should be added in one operation to leave 1·25 per cent. soda after its addition. Boiling for half an hour follows, and the fibre is filtered on a linen filter and washed free of alkali. The precipitate is then washed on the filter with boiling 1·25 per cent. sulphuric acid, thus dissolving any material precipitated by the addition of alkali, and finally washed free of acid with water. The advantage of the Sweeney method over the official method—namely, the saving of time and trouble due to two filtrations—is thus preserved, and the results as recorded show that the modification proposed yields results in agreement with the official figures within the experimental error, which is not the case with the Sweeney method.

H. F. E. H.

Solubility of Ether in Normal Salt Solution. R. R. Bennett. (*Pharm. J.*, 1912, 89, 146.)—The author has determined, by the cryoscopic method, the solubility of pure and commercial ether in "normal" salt solutions, prepared by dissolving 0·91 grm. pure sodium chloride in 99·09 grms. of distilled water. The freezing-point of this "normal" solution is −0·56° C. When commercial ether is added to the salt solution, the freezing-point of the solution is always a little lower than when the same quantity of pure ethyl ether has been added. This is due to the fact that alcohol has a smaller molecular weight than ether, and therefore produces a greater depression. It is found that there is increased molecular dissociation in the salt solution in the presence of ether.

The solubility results are shown below:

Temperature, °C.	Pure Ethyl Ether.		Purified Ethyl Ether Prepared from Methylated Spirit.	
	Grms. Ether Dissolved by 100 Grms. Normal Salt Solution.	C.c. Ether (15° C.) Dissolved by 100 C.c. Normal Salt Solution.	Grms. Ether Dissolved by 100 Grms. Normal Salt Solution.	C.c. Ether (15° C.) Dissolved by 100 C.c. Normal Salt Solution.
0°	13·08	18·27	13·46	18·80
5°	11·15	15·58	11·55	16·14
10°	9·45	13·20	9·87	13·79
15°	8·10	11·31	8·50	11·87
20°	6·87	9·60	7·38	10·31
25°	5·96	8·33	6·46	9·02
30°	5·30	7·40	5·83	8·14

The amount of ether present in solution at the different temperatures was ascertained by reference to a curve of freezing-points plotted out from actual determinations made on solutions containing known quantities of ether. A. R. T.

Examination of Methods used in the Analysis of Honey. J. Fiehe and Stegmuller. (*Arbeit. Kaiserl. Gesundheitsamte*, 1912, **40**, 305-356.)—The authors give the results of a critical examination and comparison of various methods which have been proposed and which are used for the analysis of honey. The results of their work may be summarised as follows: The total solids of honey may be estimated accurately as follows: The honey, or its solution, is mixed with sand and dried at about 65° C. in a closed vessel from which the air is exhausted, and to which a current of dry air is admitted in such quantity that the pressure in the vessel is maintained at about 30 mm. When the total solids are calculated from the sp. gr. (d) of a 20 per cent. solution of the honey, according to the formula, $\text{T. S.} = \frac{d-1}{0{\cdot}0007714}$, only approximately correct results are obtained. Litmus-paper is the best indicator to use in titrating the acidity of honey, and, as it is doubtful whether the acidity is due entirely to the presence of formic acid, the result should be expressed in c.c. of $\frac{N}{1}$ alkali solution. The estimation of the alkalinity of the ash of honey may be combined with a simple acidimetric titration of the phosphates present; the weighed ash is heated with 20 c.c. of $\frac{N}{10}$ hydrochloric acid in a covered flask on a water-bath for one hour and, after cooling, the solution is titrated with $\frac{N}{10}$ alkali solution using methyl orange as indicator. Two c.c. of a neutral 2 per cent. calcium chloride solution are now added, together with a few drops of phenolphthaleïn solution, and the titration with alkali is continued until a pink coloration is obtained; the quantity of alkali used in this second titration is equivalent to the phosphate present, each c.c. of $\frac{N}{10}$ solution corresponding with 0·0035 grm. of P_2O_5. The sugars, according to the author, should be estimated gravimetrically. As regards the tests which have been proposed for the detection of artificial invert sugar, Fiehe's reaction (ANALYST, 1908, **33**, 397) yields the most trustworthy results, especially in combination with the test for enzymes (ANALYST, 1910, **35**, 165); the presence of starch syrup may be detected by a test also described by Fiehe (ANALYST, 1909, **34**, 440). Ley's test (ANALYST, 1907, **32**, 291) was found to be unsuitable for the detection of added invert sugar, and the results obtained by Lund's test for proteins (precipitation with tannin) did not afford much evidence as to the purity of the samples of honey examined.

W. P. S.

Estimation of Lecithin in Medicinal Tablets. C. Virchow. (*Chem. Zeit.*, 1912, **36**, 906-907.)—A modification of a process described previously by the author (*cf.* ANALYST, 1911, **36**, 510) is recommended for the estimation of lecithin in tablets, in the so-called lecithin-albumin, etc. Two grms. of the substance are boiled with three successive quantities of 25 c.c. of absolute alcohol, the alcoholic extracts are filtered and evaporated together with a quantity of alcohol which is used for washing the extracted mass and the filter. The residue obtained after the evaporation of the alcohol is dried completely, and extracted with 20 c.c. of warm anhydrous ether, the ethereal extract being then evaporated, the residue weighed, and the phosphorus contained in it estimated as described (*loc. cit.*).

W. P. S.

Estimation of Dirt in Milk. W. F. Lowe. (*Chem. News*, 1912, **106**, 61-62.) —The dirt which is present in certain samples of milk usually consists of cow-dung, and several methods for its estimation have been proposed. In experiments carried out by the author it was found that when cow-dung is added to milk, then separated by sedimentation, collected on a filter, washed, and weighed, the dry residue obtained only weighs about one-eighth of the weight of moist cow-dung added. It was, however, ascertained as the result of further experiments that the volume of the sediment (in c.c.) was practically equal to the weight of the added cow-dung (in grms.), and the author, therefore, employs the following method for the estimation: Five hundred c.c. of the milk are thoroughly mixed and placed in a cylinder, the lower end of which is drawn out and attached by means of a piece of rubber tubing to a small glass tube, graduated in 0·01 c.c. The bottom of this tube may be closed with a rubber stopper. After standing for about fifteen hours, the volume of the sediment is noted; the sediment is then removed from the tube and examined microscopically. It will usually be found to consist of undigested vegetable fibre and tissue stained yellowish with bile, and the presence of bile may be proved by moistening the sediment with sugar solution, drying the mixture, and adding a drop of sulphuric acid. A red colour develops round the particles stained with bile. W. P. S.

Preparation of Milk Serums and their Use in the Analysis of Milk. R. Pfyl and R. Turnau. (*Arbeit. Kaiserl. Gesundheitsamte*, 1912, **40**, 245-304.)—A serum which contains the albumin and globulin of the milk may be obtained by shaking 50 c.c. of the milk with 5 c.c. of carbon tetrachloride for ten minutes, then adding 1 c.c. of 20 per cent. acetic acid, again shaking the mixture, and submitting it to centrifugal action. When it is desired to prepare a serum free from coagulable proteins, the milk is heated in a flask on a boiling water-bath for twenty minutes, the flask being provided with a reflux apparatus; 50 c.c. of the heated milk are then treated as described above. The serums may be employed in place of those obtained by the use of calcium chloride, etc. (*cf.* ANALYST, 1907, **32**, 117), over which they possess certain advantages. They are suitable for the determination of various physical and chemical constants in the analysis of milk, and afford a means of distinguishing raw from heated milk. The serum obtained from heated milk does not yield a precipitate when again heated. W. P. S.

Estimation of Sucrose in Condensed Milk. H. Nowak. (*Zeitsch. anal. Chem.*, 1912, **51**, 610-614.)—The method proposed recently by Jolles (*cf.* ANALYST, 1911, **36**, 28) was found to be trustworthy; it depends on the fact that all mono- and disaccharides, with the exception of sucrose, are rendered optically inactive when they are heated with sodium hydroxide solution under certain conditions. The author finds that the most convenient and satisfactory method of carrying out the process is as follows: Five grms. of the condensed milk under examination are dissolved in about 200 c.c. of water in a 250 c.c. flask, 10 c.c. of Fehling's copper sulphate solution are added, the mixture is nearly neutralised with sodium hydroxide solution (it should still have a very faintly acid reaction), then diluted to the mark, and filtered. The lactose may be estimated in a portion of the filtrate in the usual way. Another portion of about 100 c.c. of the filtrate is now treated with about 0·5 grm. (or more, if necessary) of potassium ferrocyanide, in order to precipitate the excess of copper, and again filtered. Fifty c.c. of this filtrate are placed in a Lintner's pressure flask, 1·3 c.c. of a 16 per cent. sodium hydroxide solution are added, the flask is closed, and heated for forty-five minutes in a boiling water-bath. After cooling, 3·7 c.c. of basic lead acetate solution are added, the mixture is shaken, and filtered. The optical rotation of the filtrate is then observed in a 220 mm. tube. The reading multiplied by 0·75 gives grms. of sucrose per 100 c.c. of the filtrate, and the percentage quantity of the sugar is then calculated. The addition of the alkali and basic lead acetate increases the volume of the 50 c.c. of filtrate to 55 c.c., and the reading is consequently observed in a 220 mm. tube. For example, 5 grms. of a sample of condensed milk to which 40 per cent. of sucrose had been added, when treated as described above, gave a reading of +1·06° corresponding with 39·75 per cent. of sucrose.

W. P. S.

Direct Determination of Sucrose in the Presence of Reducing Sugars. W. E. Cross and W. G. Taggart. (*Intern. Sugar J.*, 1912, **14**, 444-448; through *J. Soc. Chem. Ind.*, 1912, **31**, 831.)—As a more rapid means than the Clerget inversion process of determining the sucrose content of sugar-factory products containing reducing sugars, Jolles (ANALYST, 1911, **36**, 28) has recommended destruction of the rotatory power of the dextrose and lœvulose by sodium hydroxide. Using samples of cane sugars and molasses the authors obtained much lower results than the Pellet-Lemeland process with the Clerget-Herzfeld inversion method, the differences varying from 2 to 14 per cent., and determinations made with pure solutions of sucrose, dextrose, and lœvulose, in the proportions obtaining in cane molasses confirmed these figures. Experiments were afterwards made for the purpose of modifying the process to obtain more trustworthy results. As finally elaborated the procedure now recommended is as follows: Fifty c.c. of the "normal" sugar solution are transferred to a 100 c.c. flask, 6·3 c.c. of sodium hydroxide solution (sp. gr. 1·332) and 7·5 c.c. of hydrogen peroxide (100 vol.) added, and the mixture cooled to prevent effervescence. The flask is then immersed in a water-bath at 55° C. for twenty minutes, after which the liquid is cooled, acidified with acetic acid, made up to the mark, clarified with dry basic lead acetate, filtered, and polarised, using preferably the 400 mm. tube. It is stated that the solution thus obtained is almost colourless and quite bright. On applying (*a*) this modification, and comparing the results with (*b*) the Clerget-Herzfeld process, satisfactory results were obtained, some of these being as follows: Syrup (*a*) 32·40, (*b*) 31·87; open-kettle molasses (*a*) 41·12, (*b*) 40·98; molasses (*a*) 28·40, (*b*) 27·93 per cent. As this method has been found so reliable in the authors' hands, they recommend it as a substitute for the Clerget-Herzfeld inversion method in the analysis of syrups and molasses, and especially as a routine method for the determination of the sucrose content of low-grade products in hot-room goods.

Estimation of Essential Oil in Preparations of Mustard. H. Penau. (*J. Pharm. Chim.*, 1912, **6**, 160-164.)—Estimation of essential oil in mustard by the silver sulphide gravimetric method is unreliable, results differing by 16 per cent. being obtained with the same sample of mustard. The following modification of the French Codex method was found to be the most accurate of any: Five grms. of the mustard are digested with 100 c.c. of water in a closed flask for six hours, after which 20 c.c. of alcohol and 20 c.c. of poppy-seed oil are introduced, and the flask thoroughly shaken. The liquid is then very slowly distilled on a glycerol bath, and 90 c.c. of the distillate collected in a flask containing 10 c.c. of 50 per cent. ammonia solution. After the addition of 20 c.c. of $\frac{N}{10}$ silver nitrate solution, the flask containing the distillate is closed, and left in the dark for twenty-four hours, the precipitate being then rapidly filtered off, and washed with water. The filtrate is slightly acidified with nitric acid, the silver precipitated with 1 c.c. of $\frac{N}{10}$ hydrochloric acid, and the precipitate dried on counterpoised filters and weighed. The excess of silver may also be estimated volumetrically by the potassium cyanide method, though the results are not so good as by the gravimetric method (error in five estimations of allyl mustard oil = 2·4 per cent.). Volumetric estimation of the silver with ammonium thiocyanate is inaccurate (error in five estimations = 5·1 per cent.). (*Cf.* ANALYST, 1912, 309.)

C. A. M.

Methods of Estimating Nicotine in Tobacco and Tobacco Extracts. J. Tóth. (*Chem. Zeit.*, 1912, **36**, 937-938.)—The silico-tungstic acid method of Bertrand and Javillier (ANALYST, 1909, **34**, 219) gives results in close agreement with those given by the methods of the author and of Kissling. The following modification of the method was used: Ten grms. of the dried powdered tobacco are digested for fifteen to twenty minutes on the water-bath with 100 c.c. of 0·5 per cent. hydrochloric acid, then centrifuged and decanted, and the residue treated similarly three times. To the solutions thus obtained are added 10 to 20 per cent. of silico-tungstic acid or potassium silico-tungstate, and the precipitated nicotine silico-tungstate separated in the centrifuge. After standing for one or two days the precipitate is taken up in a little dilute hydrochloric acid, mixed with the reagent, and again separated. It is next mixed with ignited magnesia, and heated in a current of steam, and the nicotine in the distillate titrated, using iodeosin as indicator. The method is particularly useful for estimating nicotine in the presence of ammonia or pyridine bases, none of which are precipitated by silico-tungstic acid from a hydrochloric acid solution. (*Cf.* ANALYST, 1911, **36**, 106, 143, 279, 348, 544, 545.)

C. A. M.

Solidified (Hydrogenised) Oils. A. Bomer. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 104-112.)—During recent years many processes for solidifying oils have been patented, and these methods consist essentially in subjecting the oil to the action of hydrogen in the presence of a catalyst. According to the claims of the patentees the catalysts employed may be metals (such as nickel, platinum, palladium, etc.) or metallic oxides in a fine state of division. The solidifying process is carried on to any desired extent according to the consistence desired in the finished product. For instance, earthnut oil may be hydrogenised until it resembles tallow, or, by a shorter period of treatment, it may be converted into a product which cannot be distinguished from lard as regards its appearance, consistence, taste, smell. The process results in the conversion of the unsaturated fatty acids (oleic acid, etc.) of the oil into stearic acid; the iodine value is diminished to a very considerable extent, and the melting and solidifying points raised. For example, solidified earthnut oil may have an iodine value as low as 42·2 and sesame oil 25·4, these being the results obtained by the author on the examination of some of the products. The phytosterol present in vegetable fats is not altered by the treatment, and the phytosterol acetate test described previously by the author enables a solidified vegetable oil to be distinguished from an animal fat yielding similar chemical and physical constants. W. P. S.

Estimation of Succinic and Malic Acids in Wine. C. von der Heide and E. Schwenk. (*Zeitsch. anal. Chem.*, 1912, **51**, 628-638.)—Slight modifications are made in the methods described previously by von der Heide and Steiner (*cf.* ANALYST, 1909, **34**, 278, 279). In the case of succinic acid, the ethereal solution obtained at the end of the process (*loc. cit.*) is evaporated after the addition of water, and the residual aqueous solution is rendered alkaline with barium hydroxide. The mixture is then heated on a water-bath for ten minutes, the excess of barium is removed as carbonate, the mixture is cooled, filtered, and the filtrate titrated with silver nitrate solution. It is recommended that at least two hours should elapse after the addition of the silver nitrate, and before the excess of the latter is titrated. For the estimation of malic and succinic acids together, 100 c.c. of the wine are evaporated to a volume of 20 c.c., and in this residual liquid the tartaric acid is separated and estimated. The filtrate from the potassium hydrogen tartrate precipitate is treated as described (*loc. cit.*), but, after the ether has been evaporated from the ethereal extract, the residue is treated with barium hydroxide, and the solution containing soluble barium salts of the two acids is evaporated, the residue ignited, and titrated. The alkalinity gives the sum of the quantity of succinic and malic acids present. W. P. S.

Estimation of Phosphoric Acid in Wines. C. von der Heide and J. Schwenk. (*Zeitsch. anal. Chem.*, 1912, **51**, 615-627.)—As regards the alleged loss of phosphoric acid during the ignition of the total solids of wine, the authors find that no such loss takes place, and that in the case of ordinary table wines, sweet wines, musts, and yeast, the addition of sodium carbonate and potassium nitrate previous to the ignition is quite unnecessary. In preparing the ash of the wine for the estimation of the phosphoric acid, it is recommended that 50 c.c. of the sample be evaporated, and the residue incinerated; the carbonaceous mass is then extracted with water, and the washed carbon is ignited to a white ash, which is dissolved in nitric acid and the solution added to the aqueous extract. This solution is used for the estimation of the phosphoric acid, for which purpose the method proposed by von Lorenz (*Landw. Versuchsst.*, 1901, **55**, 183; *cf.* ANALYST, 1903, **28**, 229) is recommended. Grete's method (ANALYST, 1909, **34**, 541) is also trustworthy, and may be employed for estimating the total phosphoric acid directly—that is, without previously incinerating the wine residue. The amount of organically combined phosphoric acid in wine is only a small proportion of the total phosphoric acid; it may be estimated by the method described by Philippe and Duperthius (*Mitt. a. d. Geb. der Lebensmittelunters. u. Hyg.*, 1910, 1, 125), which depends on the precipitation of the inorganic phosphoric acid by means of barium hydroxide, the barium salts of the organically combined phosphoric acid being soluble in water.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Studies of Amylases. IV. A Further Investigation of the Properties of Pancreatic Amylase. H. C. Sherman and M. D. Schlesinger. (*J. Amer. Chem. Soc.*, 1912, 34, 1104-1111.)—In a previous paper (*J. Amer. Chem. Soc.*, 1911, 33, 1195) the authors describe preparations of purified pancreatic amylase, having an activity of about 5,000 on Lintner's scale for purified diastases. A detailed method of preparing such amylase is described with modifications suggested by recent work, the preparation having a heat of combustion of 5,568 calories per grm., and the following approximate composition, C 51·9, H 6·6, N 15·3, S 1·0, P 0·8, O (and undetermined) 24·4. Coagulation occurs at 70° C. In the determination of diastatic activity it was at first found that the yield of reducing sugar did not amount to more than 85 per cent. of the starch used, but on increasing the amount of enzyme and allowing it to act on relatively small amounts of starch, such as 20 mgrms. of enzyme to 100 c.c. of 1 per cent. soluble starch for seventy-two hours, the resulting sugars were found to be a mixture of maltose and glucose, and confirmed by a special quantitative modification of the osazone method. The method of purification of the pancreatic-amylase had thus resulted in a concentration of a glucase or maltase in addition to the amylase. In the absence of electrolytes, the preparations very rapidly lose their enzymic activity, and even in the presence of salt and phosphate, as used for the determination of diastatic power, the purified amylase deteriorated from 33 to 50 per cent. in twenty-four hours, the commercial pancreatins being much more stable. This was thought to be due to the presence of a protease of a trypsin-like character, which might digest the protein with which the amylase was associated. It was found on measuring the proteolytic activity of both the crude pancreatin and the purified amylase prepared from it, that the increase in activity of both enzymes was about proportional. Other preparations examined showed much higher proteolytic activity with an amylolytic action only one-fifth as great. It is possible that one of the pancreas enzymes is both a protease and amylase, with the potentiality of an increase in the one activity accompanied by a decrease in the other.

H. F. E. H.

Note by Abstracter.—The formation of glucose, if it occurs, of course vitiates the Lintner value for diastatic activity, since this value is an expression of the activity of the enzyme in terms of maltose produced. The fact that in some cases the "R" (= per cent. of maltose) does not exceed 100 does not rule out the possibility of glucose having been formed simultaneously with or at the expense of the maltose.

Chemical Detection of Blood in Blood-Stains. E. Schär. (*Pharm. J.*, 1912, 89, 157-158.)—In cases where the quantity of suspected material is too small for spectroscopic examination, or for the preparation of hæmin crystals, the author employs several of the well-known colour-reactions based on the action of blood upon hydrogen peroxide in presence of oxidisable substances forming coloured oxidation-products. The blood, in solution, is mixed with the solution of oxidisable substance and either hydrogen peroxide, or turpentine oil previously shaken with water in the presence of air. The tests are: (1) tincture of guaiacum, which yields a blue colour; (2) tincture of aloes (isobarbaloin), a raspberry-red colour; (3) Adler's test with an acetic benzidine solution, a greenish-blue; (4) Adler and v. Fürth's leucomalachite green test, giving a dark green; (5) and the author's test with hydrocœrulignone, yielding the scarlet-coloured cœrulignone. Certain inorganic substances (iron and copper salts) and substances containing enzymes of the "peroxydase" class give similar reactions, but these may be ruled out, in the first instance (inorganic substances), by control reactions, and secondly, as regards interfering enzymes, by first heating the material for a short time from 80° to 100° C. In any case, a negative result may be considered as proof of the absence of blood.

According to the author, the best method of preparing the blood solution for the tests, whether from old or recent stains, is by means of chloral hydrate. The spot or stain is first moistened with a drop of acetic acid, and then soaked for one to several hours in chloral hydrate solution of 70 to 80 per cent. strength. The blood solution may then be used for the above tests. Zone reactions may be carried out by dissolving a little of the solutions of one of the oxidisable matters in the chloral hydrate solution of the blood, and carefully adding a quantity of hydrogen peroxide solution (3 per cent.), diluted with twice its volume of alcohol and slightly acidulated with acetic acid, by superposition. A deeply-coloured zone will appear between the two strata in the presence of blood, and will gradually spread into the liquids. When pyridine is used for the extraction of blood from stains, etc., the colour-reaction is accelerated and intensified. A. R. T.

Physico-Chemical Method for Determining the Changes taking place in Meats. O. von Fürth and E. Lenk. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, 24, 189-197.)—When flesh is immersed in a 15 to 25 per cent. sodium chloride solution it gradually diminishes in weight, but this only holds good for flesh one day old, counting from the death of the animal from which it was obtained. In the case of flesh more than one day old, an increase in weight is observed during the first ten to fifteen hours it is immersed in the salt solution, and this increase is always followed by a decrease during the succeeding hours. Chilled beef rapidly increases in weight when placed in sodium chloride solution. The increase in weight is probably due to the formation of lactic acid, as acids, when not above certain concentrations, greatly accelerate the imbibition capacity of muscular tissue, etc.; the subsequent decrease in weight appears to be due to a coagulation of the proteins. It is suggested that when the subject has been further investigated the phenomenon may be of use in testing meats. W. P. S.

Quantitative Estimation of Quinine in Urine and in Blood. A. Baldoni. (*Arch. d. Farmacol. sperim.*, 1912, 13, 324-352; through *Chem. Zentralb.*, 1912, II.

554.)—An experimental examination of the methods of Gordin (*Ber.*, 1899, 32, 2843), Kleine (*Zeitsch. f. Hyg.- u. Infect.- Krankh.*, 1901, 38, 458), Gaglio (*Atti per gli studi del. Malaria*, 1905, 6, 77), and Nishi (*Arch. f. exp. Pathol. u. Pharmak.*, 1909, 60, 312). The method of Nishi was found to give results which were uniformly somewhat low, whilst the method of Kleine showed errors somewhat beyond the permissible limit, and not all in the same direction. The results obtained by the methods of Gordin and Gaglio agreed closely with each other and with the amount of quinine actually present. Making use of these methods, the author was able to show that the resorption of quinine is more rapid when it is administered by the mouth than when administered hypodermically. Quinine disappears from the blood in a very few hours, and in no case could it be detected twenty-four hours after administration, a statement which conflicts with that of De Luca (*Arch. d. Farmacol. sperim.*, 1901, 2, 390). G. C. J.

Iodine Content of Thyroid Glands. N. H. Martin. (*Pharm. J.*, 1912, 89, 144-145.)—The following analyses of sheeps' thyroid glands, normal in size, are given:

Date.	Number of Glands Used.	Weight of Fresh Glands.	Weight of *Thyroideum Siccum* Obtained.	Average Weight of Each Fresh Gland.	Average Yield of *Siccum* per Gland.	Iodine in [illegible]	Iodine on Fresh Weight.	Average Iodine per Gland.
1911.		Grms.	Grms.	Grms.	Grms.	Per Cent.	Per Cent.	Grms.
July ...	590	787	195	1·33	0·33	0·40	0·099	0·00132
August ...	885	1,096	235	1·24	0·27	0·36	0·077	0·00095
September...	490	631	200	1·29	0·41	0·34	0·107	0·00138
October ...	490	584	162	1·19	0·30	0·38	0·105	0·00125
November...	190	243	54	1·28	0·28	0·30	0·066	0·00085
December ...	253	408	72	1·61	0·28	0·34	0·060	0·00097
1912.								
January ...	216	320	64	1·48	0·29	0·32	0·064	0·00095
February ...	184	289	60	1·57	0·33	0·32	0·066	0·00104
March ...	393	618	171	1·57	0·44	0·34	0·095	0·00149
April ...	1,061	1,580	611	1·46	0·57	0·30	0·116	0·00169
May ...	958	1,459	487	1·52	0·51	0·34	0·113	0·00172
June ...	830	1,284	426	1·55	0·51	0·38	0·126	0·00195
	6,560	9,299	2,737	1·42	0·38	0·34	0·091	0·00129

The author has examined glands having the enormous weights of 12·0, 14·0, and 32·5 grms., containing 0·22 per cent., 0·20, and 0·08 per cent. of iodine in the *thyroideum siccum*, and 0·00616, 0·00660, and 0·00600 grms. iodine per gland respectively. Thus the larger amount of iodine in these glands was not proportional to their increased weight, and the percentage of iodine in the dry substance was reduced.

The figures, as a whole, lend no support to the statement that the amount of iodine in the dry substance is greater in the winter months. A. R. T.

Measurement of Relative Tryptic Activity. A. R. Smith. (*Pharm. J.*, 1912, 89, 137-139.)—The author has applied the method of Sorensen (*Compt. rend. Trav. du Lab. de Carlsberg*, 1907, 7, 1) to the determination of the relative tryptic activity of certain pharmaceutical preparations. The method is based on the facts that the main products of the hydrolysis by trypsin are amino-acids, and that if the basicity of the amino-groups be neutralised, the carboxyl-groups can be titrated in the usual way. Schiff has shown that these amino-groups can be converted into neutral methylenic groups by adding formaldehyde, and the acids can then be estimated by titration. The reaction with formaldehyde is reversible in presence of water, but an increase of formaldehyde or decrease in the amount of water present tends to make the reaction proceed in the desired direction.

The tests are carried out as follows: A 4 per cent. solution of casein is prepared by dissolving the substance in recently-boiled and cooled water containing 30 c.c. of $\frac{N}{1}$ sodium hydroxide per litre. The solution is made neutral to phenolphthalein with alkali before making up to volume, and a little chloroform is added as an antiseptic. Twenty-five c.c. of this casein solution at 55° C. is mixed with a known volume of the trypsin-containing solution at the same temperature, and water added (55° C.) to make the volume to 50 c.c. One grm. of the enzyme preparation to be tested is triturated in a mortar with a little chloroform-water, and the solution made up to 100 c.c. After standing one hour, 1 c.c. of the well-mixed, unfiltered solution is added to the 25 c.c. of casein solution at 55° C., and the mixture diluted as stated. The mixed solutions are kept at this temperature for twenty minutes, when 20 c.c. of the liquid is removed, and titrated with $\frac{N}{5}$ sodium hydroxide solution after first adding 10 c.c. of neutral formaldehyde solution. (This solution is prepared immediately before use by mixing 1 c.c. of a $\frac{1}{2}$ per cent. solution of phenolphthalein in 50 per cent. alcohol with 50 c.c. of 40 per cent. formaldehyde solution, and adding $\frac{N}{5}$ sodium hydroxide till the colour is just pink.) Sorensen recommends that the titration of the "digest" solution should be carried on till the liquid is the same colour as a control prepared by adding 10 c.c. of the formaldehyde reagent to 20 c.c. of water and 5 c.c. of $\frac{N}{5}$ caustic soda. The solution is titrated back with $\frac{N}{5}$ hydrochloric acid till just pink, when 1 drop of standard alkali makes it distinctly pink, and 2 drops, red. This is the end-point.

A blank experiment must be made by titrating 20 c.c. of the mixed casein and trypsin solutions before digesting, in order to ascertain the volume of alkali required. The difference after digestion is that due to the acids produced in the hydrolysis. The figure multiplied by 100 may be used to express the relative activity of trypsin preparations.

Further experiments with the method showed: (1) That relative measurement of activity can be made so long as not more than 3·5 to 3·7 c.c. of $\frac{N}{5}$ alkali are used in the titration of 20 c.c. of the "digest"; (2) that 55° C. is the optimum temperature for digestion; (3) that under conditions (1) and (2) a period of twenty minutes for digestion is convenient, since then the relative hydrolysis is in direct proportion to the trypsin present. The following figures were obtained by using this method on certain preparations:

Sample.	C.c. NaOH $\frac{N}{5}$.		
	Titration.	Blank.	Tryptic Activity.
Pancreatin 1	3·45	2·08	1·37
„ 2	2·76	2·04	0·72
„ 3	4·02	2·05	1·97
„ 4	3·64	2·08	1·56
„ 5	3·42	2·05	1·37
Trypsin 1	3·34	2·06	1·28
„ 2	3·78	2·06	1·72

A. R. T.

ORGANIC ANALYSIS.

The Coagulation of Albumin by Heat and its Precipitation by Mercuric-Potassium Iodide. Gravimetric and Volumetric Estimations. L. Vallery. (*Compt. rend.*, 1912, **155**, 417-420.)—In a previous note (*Compt. rend.*, 1911, 153), the author showed that the coagulation of albumin in urine by heat in presence of acetic or trichloracetic acid is incomplete, and he described a new gravimetric method for its estimation, based on the use of Tanret's or Esbach's reagent, giving distinctly higher results. It is now shown that the deficiency in the quantity of albumin coagulated by heat is due to the partial hydrolysis of the albumin by the action of the acid, and the loss when acetic acid is used is always less than when trichloracetic acid is present, owing to the greater degree of dissociation of the latter. It is now found that if a non-dissociated acid—*e.g.*, normal caproïc acid—be employed, perfectly satisfactory results are obtainable without appreciable hydrolysis. A few drops of this acid are to be added to the sample after this has been neutralised with sodium hydroxide in presence of phenolphthaleïn, but without the addition of an electrolyte. This procedure is especially recommended in the analysis of urine. The volumetric estimation of albumin by the use of Tanret's reagent depends on an adsorption reaction with mercury which can be represented by a hyperbolic formula. In the case of serum and egg albumins this law is expressed by the equation $\frac{1}{x}\log.\frac{H}{H-h}=K$; where x = the quantity of albumin, H = the initial quantity of mercury, h = the quantity of mercury adsorbed, and K is a constant. By making H = 0·20 grm. and diluting to 200 c.c. after precipitation, the value of K is 0·54 for serum albumin and 0·52 for egg albumin. The presence of urea and sodium chloride is without influence on the curve, but the presence of phosphates lowers the constant considerably, to an extent depending on their quantity. For this reason the volumetric process is not at present applicable for urine and other organic fluids containing more phosphates than serum and egg albumins.

J. F. B.

Benzoic Acid as an Acidimetric Standard. G. W. Morey. (*J. Amer. Chem. Soc.*, 1912, **34**, 1027.)—A hydrochloric acid solution was standardised by the distillation method of Hulett and Bonner (*J. Amer. Chem. Soc.*, 1909, **31**, 390), by the gravimetric silver chloride method, against sulphuric acid standardised gravimetrically by precipitation as barium sulphate and volumetrically against sodium oxalate, and was finally standardised against benzoic acid. The methods used are described in detail. The benzoic acid was prepared by recrystallising pure samples, twice from alcohol and once from water, subliming in vacuo, fusing at 140° C., and casting into sticks. About 1 grm. was weighed, placed in a 300 c.c. flask, swept free from carbon dioxide, dissolved in 20 c.c. of alcohol, and titrated with $\frac{N}{10}$ alkali, 3 drops of 1 per cent. phenolphthaleïn being used as indicator, and air, free from carbon dioxide, being led through the liquid. A correction for the effect of the alcohol on the end-point was applied. The appended table shows the accuracy of the benzoic acid method:

Method.	$\frac{N}{10}$ factor for HCl.
Direct by Hulett	0·9980
Direct by AgCl	0·9984
H_2SO_4—$BaSO_4$—NaOH—HCl	0·9984
H_2SO_4—$Na_2C_2O_4$—NaOH—HCl	0·9982
$C_6H_5CO_2H$—$Ba(OH)_2$—HCl	0·9984
$C_6H_5CO_2H$—NaOH—HCl	0·9981

Its stability and lack of hygroscopicity make it convenient, and the method involves only a single weighing and a titration. O. E. M.

Estimation of Carbon by Moist Combustion. H. Simonis and F. H. Thies. (*Chem. Zeit.*, 1912, **36**, 917-918.)—The following method is recommended for the estimation of carbon in such substances as phenylacetaldehyde, etc., the oxidation being carried out by means of potassium bichromate and sulphuric acid. A portion of about 0·2 grm. of the substance is weighed into a small platinum boat, which is then filled with powdered potassium bichromate and introduced into a round-bottomed flask of about 200 c.c. capacity. A further quantity of 20 grms. of bichromate are placed in the flask, which is then closed with a stopper through which pass the stem of a tapped funnel and a delivery-tube. The latter connects with a wide tube filled with glass-wool in which most of the moisture leaving the flask is condensed; the other end of the wide tube is in communication with a combustion-tube filled with copper oxide and heated by suitable means. The necessary drying and absorption apparatus are attached to the end of this combustion-tube. It is essential that the gases leaving the oxidation-flask be passed over hot copper oxide, as they contain small quantities of unoxidised carbon compounds. At the commencement of the operation, a soda-lime tube is attached to the combustion-tube in place of the absorption apparatus, and a current of oxygen is passed through the whole apparatus, the supply of oxygen being introduced through a T-piece and narrow tube which extends through the delivery-tube to the bottom of the flask; oxygen is also supplied to the top of the funnel which contains sulphuric acid. When the apparatus has been filled with oxygen the absorption apparatus is connected, and the sulphuric acid is admitted from the funnel, drop by drop, or at such a rate that the evolved gases pass through the absorption vessels at the rate of a few bubbles a second.

The contents of the flask are eventually heated with a burner for thirty minutes, and pure air is passed through the apparatus before the absorption vessels are weighed. Duplicate estimations of carbon in phenylacetaldehyde yielded 79·98 and 79·96 per cent. of carbon, respectively, against 80 per cent. required by theory. W. P. S.

Methods for Determining Neo- and Allo-Chlorophyll in the Presence of one Another. C. A. Jacobson and L. Marchlewski. (*Amer. Chem. J.*, 1912, **48**, 111-124.)—The authors have shown previously (*Amer. Chem. J.*, 1911, **47**, 221) that the ratio of neo-chlorophyll to allo-chlorophyll in plants is not a constant one, varying not only in different species, but in the same when the samples have been grown under different conditions.

Method 1.—Using ultra-violet spectrum.

Neo-chlorophyllan was prepared by two methods in order to obtain a photograph of the ultra-violet spectrum of this material in the pure state. By the shorter method the starting-point is chlorophyllan and the neo-chlorophyllan so obtained is a black, amorphous substance something like oak bark. It gives a deep, olive-green solution, thus distinguishing it from allo-chlorophyllan which has a reddish-brown colour. The ultra-violet spectrum in a concentration of 0·00004 grm. per c.c. of chloroform shows three absorption bands, the wave-lengths in a layer of 6 mm. thickness being (1) λ 365·0 to 382·7, (2) λ 388·8 to 405·0, (3) λ 407·4 to 426·5.

By the second and longer method of preparation, the neo-chlorophyllan is extracted directly from leaves by the use of organic solvents alone and remains in solution in carbon disulphide, while the allo-chlorophyllan is removed by continuous alcoholic extraction. The ultra-violet spectrum is identical with that obtained by the first method. A series of eleven photographs is supplied showing the ultra-violet spectra of artificial mixtures of the neo- and allo- varieties in different proportions equally graded, and the method consists in preparing the chlorophyllan of the plant to be examined in the ordinary way, and then to take a photograph in the ultra-violet of the spectrum yielded by a solution whose concentration is equal to those photographed in the series. A direct comparison with the plates supplied will then reveal the composition of the mixture to within 2 per cent. Care must be taken first to remove all the yellow colouring matters by means of several precipitations with alcohol from the concentrated chloroform solution before photographing the spectrum, except in the case of Acer platinoides, whose yellow colouring matter was found not to interfere.

Method 2 is based on the extinction co-efficients of the chloroform solutions, and has already been employed by one of the authors for determining the total green colouring matter in a plant. A cadmium lamp was employed, giving true monochromatic light in the green part of the spectrum. A table is given of the same eleven artificial mixtures as given above with their extinction co-efficients, for cadmium light λ = 508 μμ and sodium light λ = 589 μμ. The values for both lights are plotted and yield practically straight line curves, and show that the percentage composition of any mixture of neo- and allo-chlorophyllan is a linear function of its extinction co-efficient. The first method is less sensitive to impurities, is more adapted to practical application, and can be used in very small quantities, but where sufficient material is available to allow of very thorough purification, the extinction method allows of greater accuracy. H. F. E. H.

Estimation of Cineol in Volatile Oils. F. D. Dodge. (*J. Ind. and Eng. Chem.*, 1912, **4**, 592-593.)—The author oxidises the terpenes and unsaturated compounds in the cold with potassium permanganate, and measures the remaining insoluble cineol. For the test 10 c.c. of the oil (eucalyptus, cajeput) are treated in a flask of 500 c.c. capacity with potassium permanganate solution (5 to 6 per cent.) added in small quantities, with shaking, till the reagent is no longer reduced. The flask must be kept in ice-cold water throughout the addition of the permanganate, of which from 100 to 400 c.c. are required. When excess has been added, the mixture is kept cold for twelve to eighteen hours with occasional shaking. The precipitated manganic oxide is then dissolved by sulphurous acid and the flask filled with water, the unoxidised oil collecting in the narrow neck. The oil is then transferred by a capillary pipette to a graduated tube, washed with a little alkali and its volume read off. The cineol should have the following characters: Sp. gr. 15° C., 0·929 to 0·930; be optically inactive; soluble in $3\frac{1}{2}$ volumes of 60 per cent. alcohol at 25° C. The isolation of the cineol itself allows of a determination of its purity, or of a detection of other compounds when present with it. Thus camphor, fenchone and bornyl acetate increase the sp. gr. of the residual oil and show optical rotation; inactive camphor increases the sp. gr.; petroleum products lower the sp. gr. and diminish the solubility; borneol and menthol are oxidised to camphor and menthone respectively; and menthone lowers the gravity and shows optical activity. A. R. T.

Detection and Estimation of Halides in the Presence of Cyanides by Means of Formaldehyde. K. Polstorff and H. Meyer. (*Zeitsch. anal. Chem.*, 1912, **51**, 601-609.)—The method is based on the fact that formaldehyde combines with potassium cyanide to form a compound which does not reduce silver nitrate or yield a precipitate with the latter in the presence of nitric acid; Romijn has previously made use of this reaction for the estimation of formaldehyde (ANALYST, 1897, **22**, 221). For the detection of chlorides in mixtures of the same with cyanides, a solution of the salt is rendered alkaline with potassium hydroxide and formaldehyde is added in excess; the solution is then acidified with nitric acid and tested with silver nitrate when the formation of a white, curdy precipitate which is soluble in ammonia indicates the presence of chlorides. For the estimation of chloride, bromide, or iodide in the presence of cyanide, a quantity of about 0·6 grm. of the sample is dissolved in 100 c.c. of water, the solution is rendered alkaline with potassium or sodium hydroxide, and 30 drops of a 35 per cent. formaldehyde solution are added. After the lapse of a few minutes, the mixture is acidified with nitric acid, and the halide is titrated according to Volhard's method. The cyanide may be estimated in a separate portion of the sample by Liebig's method. W. P. S.

Estimation of Halogens in Organic Compounds, including a Device for the Combustion of Volatile Substances. E. E. Reed. (*J. Amer. Chem. Soc.* 1912, **34**, 1033.)—The substance containing halogen is subjected to combustion in a quartz tube, for the arrangement of which the original should be consulted, and the halogen arrested by means of silver powder and silver oxide. The silver is weighed in a silver boat, and the separately weighed silver oxide (0·2 to 0·5 grm.) then added. The silver oxide is prepared from silver nitrate, washed, dried at 100° C., well mixed, and sealed up in 0·5 to 1·0 grm. portions. The loss on reduction is determined by heating a portion in hydrogen from 160° to 170° C. for thirty minutes. It is higher than the theory demands, on account of retention of water by the silver oxide; this is of no consequence if it is uniform. During the combustion the silver and silver oxide is maintained at about 270° C., and when it is finished, reduced by hydrogen at 170° C. In the calculation the loss in weight on reduction of the quantity of oxide used is deducted from the original weight of the boat. In the device for the combustion of volatile liquids (*cf.* original) the substance sealed in a bulb is placed in the larger limb of a U-tube communicating with the heated combustion tube into which its vapour is swept, after the bulb has been broken, by means of a current of air or nitrogen. A table shows that analyses are consistent and sufficiently accurate, and that 0·2 to 0·25 grm. of substance, but not more, may be burnt in twenty minutes. O. E. M.

Analysis of Hydrocarbon Mixtures. O. Routala. (*Ann. Acad. Sci. Fennicæ*, 1912, Ser. A, **2**, 19; through *Chem. Zentralbl.*, 1912, II., 638.)—The iodine value for mineral oils and their derivatives has only a significance when accompanied by an exact description of the conditions under which the estimation was carried out. In the estimation of ethylenic hydrocarbons in mixtures, the results deduced from the iodine value vary according to the method employed, the time of action, the temperature, and the quantity of iodine solution; these should always be specified. The bromine value, estimated according to Frank's method, affords a quantitative measure of the ethylenic hydrocarbons, and is particularly to be recommended when the quantity of the sample is small, for the determination of the degree of refining of colourless or slightly coloured mineral oil products, and for testing the purity of particular fluid ethylenic hydrocarbon preparations. The percentage of ethylenic hydrocarbons can be calculated direct from the bromine value by the formula $A = [K + (n - 5)c].G$, in which A = the percentage of ethylenes; K = a constant; n = the mean number of carbon atoms (not less than 5) in the ethylenes corresponding to the particular limits of boiling-point; and G is the bromine value. $G = \frac{100\,g}{S} = \frac{100\,v.\,0{\cdot}008}{S}$, in which g is the quantity of bromine absorbed in grms., S is the quantity of the substance taken in grms., and v = c.c. of $\frac{N}{10}$ bromine solution. The constants K and c are calculated for each hydrocarbon, so that $\frac{\text{percentage of ethylenes}}{\text{bromine value}}$ = a constant. K is 0·4375 for amylene, 0·5950 for hexylene, 0·6075 for heptylene; for each increment of CH_2 the constant increases on the average by 0·0875 = c. For instance, in a mixture of amylene and isopentane of sp. gr. 15° C. = 0·6424, a quantity of 0·2277 grm. of the sample required 39·5 c.c. of a $\frac{N}{10}$ bromide-bromate solution, corresponding to 0·3160 grm. of bromine; the bromine number is thus 138, and the sample contains 0·4375 × 138 = 60·37 per cent. of amylene. The accuracy of the results obtained by the bromine method is confirmed by those of the sulphuric acid method. J. F. B.

Relationship between the Iodine Value and the Structure of Acids of the Oleic Series. G. Ponzio and C. Gastaldi.

(*Gazz. Chim. Ital.*, 1912, **42**, 92-95.)—While the iodine value of ordinary oleic acid as estimated by the usual methods is in close agreement with the theoretical value, the results obtained with crotonic acid and certain other fatty acids are much too low. Thus in the estimation of the iodine value by the methods of Hübl (4 hours' absorption), Wijs (30 minutes) and Hanus (30 minutes) the following results were obtained:

	Hübl.	Wijs.	Hanus.	Theoretical Value.
Undecylenic acid, $CH_2{:}CH.(CH_2)_8.COOH$, ..	135·1	137·3	—	137·8
Crotonic acid, $CH_3.CH{:}CH.COOH$	17·4	10·3	4·3	295·0
2—3 Hypogæic acid, $CH_3(CH_2)_{12}.CH{:}CH.COOH$	6·6	20·4	1·9	99·8
2—3 Oleic acid, $CH_3(CH_2)_{14}.CH{:}CH.COOH$ ...	8·7	18·0	3·0	89·7

From these and similar results the conclusion is drawn that it is the position of the double bond in unsaturated acids of the series $C_nH_{2n}-2O_2$ that influences the amount of the iodine value. If the double bond is distant from the carboxyl group, as in ordinary oleic and undecylenic acids, the iodine value will be normal, whilst if it adjoins the carboxyl group, as in the case of the other acids in the table above, the iodine value will be lower than the theoretical value. Further experiments showed that it was only the velocity of the reaction that was affected by the position of the bond; for by prolonging the time of absorption to seventy hours, 2—3-oleic acid gave an iodine value of 45·9 by Hanus' method, and of 86·8 by the method of Wijs. The absorption was due to addition and not to substitution. It thus appears that an estimation of the iodine value is a good method of establishing the position of the double bond in an unsaturated acid. C. A. M.

Proximate Analysis of Complex Lipoids by Alcoholysis. Fourneau and Piettre.

(*Bull. Soc. Chim.*, 1912, **11**, 805-810.)—The authors have studied the separation of complex fatty bodies into their proximate constituents by the action of alcoholysis. The material used was lecithin containing 1·76 per cent. of nitrogen and 3·86 per cent. of phosphorus. A solution of 50 grms. of lecithin in 100 grms. of methyl alcohol was saturated at a temperature of 0° C. with gaseous hydrogen chloride. The solution was then heated at about 50° C. for one hour until the excess of hydrochloric acid had disappeared. The liquid then separated into two layers, the upper containing the esters of the fatty acids, and the lower containing the glycerophosphoric acid and choline hydrochloride. The upper layer was carefully decanted off, and washed with a little water, the washings being added to the aqueous acid layer, which latter was then exhausted with ether to remove the last traces of fatty matters. The ethereal extract was added to the fatty esters previously separated.

dried first over sodium sulphate, then over sodium carbonate; the ether was evaporated and the residue distilled under a pressure of 18 mm. Between 200° and 203° C., 18 grms. of a liquid partially crystallising at about 12° C. was distilled over. A second fraction of 13 grms. distilled between 207° and 210° C. Lastly, 3·5 grms. came over between 210° and 230° C. without any definite boiling-point, and the residue amounted to 2·5 grms. The acid solution containing the glycerophosphoric acid and the choline was concentrated at a low temperature *in vacuo* to expel most of the hydrochloric acid; the residue was treated with 500 c.c. of water and 10 grms. of animal charcoal. The solution was neutralised with chalk, made faintly alkaline with milk of lime, and, lastly, very slightly acidified with hydrochloric acid. The filtered solution was evaporated at a temperature not exceeding 55° C. The residue was taken up with absolute alcohol, which dissolves the choline hydrochloride and the calcium chloride, leaving the calcium glycerophosphate undissolved. This latter, after drying, weighed 12 grms. The alcoholic solution was evaporated, the residue dissolved in water, and the solution treated with exactly the quantity of sodium carbonate necessary to precipitate the calcium. The filtrate was evaporated and the choline hydrochloride extracted from the residue by absolute alcohol, the yield on evaporation being 7 grms. The products obtained by the quantitative alcoholysis of the lecithin were thus: 68 per cent. of stearo-oleic methyl esters, 24 per cent. of calcium glycerophosphate, and 14 per cent. of choline hydrochloride, whereas the theoretical values are respectively 76, 26, and 17 per cent. The fatty esters consisted of methyl stearate and oleate, with possibly a little linoleate. The calcium glycerophosphate was optically inactive, and could be split up into two fractions, one of which was crystalline and anhydrous. Alcoholysis is the best method of preparing cholesterin, which body, if present, separates in a remarkably pure crystalline state after the alcoholysis. The method has been applied to the investigation of the complex fats of yolk of eggs, suprarenal capsules, and sheep's skin. J. F. B.

Volumetric Method for the Determination of Phenol-*p*-Sulphonic Acid. C. E. Smith and H. C. Frey. (*J. Amer. Chem. Soc.*, 1912, **34**, 1040.)—A quantity corresponding to 0·18 to 0·2 grm. of phenol-*p*-sulphonic acid, is dissolved in 50 c.c. of water in a 250 c.c. stoppered flask with a long, narrow neck. To this are added 50 c.c. of a water solution containing 2·7833 grms. of potassium bromate and about 40 grms. of potassium bromide per litre, and 5 c.c. of hydrochloric acid of sp. gr. 1·13 to 1·19, the flask quickly stoppered to prevent loss of bromine, and allowed to stand at 20° to 25° C. for not less than ten or more than fifteen minutes. Potassium iodide solution, 1 grm. in 5 c.c. of water, is added, by pouring a small quantity round the stopper, which is then raised while the rest is quickly introduced. The solution is shaken and titrated with $\frac{N}{10}$ sodium thiosulphate. No indicator is needed, but starch can be used. The number of c.c. required is subtracted from 50; each c.c. of the remainder corresponds to 0·00435 grm. of phenol-*p*-sulphonic acid. The limit of error is ± 0·5 per cent. If accurate results are required, a rough preliminary determination is necessary when the approximate quantity of phenolsulphonic acid is unknown. O. H. M.

Parchment Paper. A. Burr, A. Wolff, and F. M. Berberich. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 197-227.)—Analyses of some fifty-eight samples of commercial parchment paper yielded the following results: The moisture varied from 5·49 to 13·10 per cent., but thirty-four of the samples contained less than 8 per cent. The amount of ash present in the papers ranged from 0·23 to 17·10 per cent., but in only twenty-one samples did it exceed 4 per cent.; in a few cases lead was detected in the ash. The papers yielded from 0·05 to 31·10 per cent. of water-soluble matters, thirty-four of the samples giving more than 10 per cent. One sample of paper contained 25·78 per cent. of sugar, and only sixteen were free from this constituent. There was evidence that magnesium chloride and calcium chloride were present in some of the papers. Experiments were also carried out with the object of ascertaining the suitability of parchment paper as a wrapper for butter, etc., particular attention being paid to the behaviour of those papers which contained an undue proportion of foreign substances. Parchment paper containing a large quantity of sugar favours the growth of moulds when wrapped round fresh butter, especially when the latter contains a large amount of water or butter-milk. The presence of salt (1 per cent.) in the butter hinders the growth of the moulds. When butter has to be kept in parchment-paper wrapping for some length of time, it is recommended that the paper before use be washed with hot water and then dipped in a salt solution. Although moulds do not grow well in the presence of glycerol, they are scarcely affected by the quantities of this substance which are present in parchment papers. A satisfactory paper for use as a butter wrapper would probably be one containing not more than 8 per cent. of water-soluble constituents, and yielding not more than 3 per cent. of ash. W. P. S.

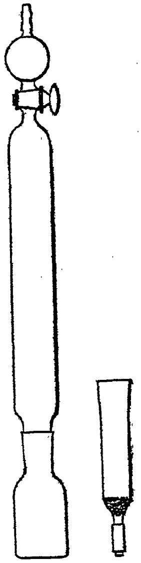
Hübener Bromide Method for the Estimation of Rubber. G. Hübener. (*Gummi-Zeit.*, 1912, **26**, 1284; through *Chem. Zentralbl.*, 1912, II., 762.)—The author replies to criticisms, especially by Hinrichsen and Kindscher (Analyst, 1912, 202), of his bromide method (*ibid.*, 1909, **34**, 365), and mentions a case in which a rubber, stated to consist of 90 per cent. caoutchouc and 10 per cent. sulphur, in reality contained only 65 per cent. caoutchouc, as the bromide method had shown. Sources of error are mentioned. The substance must be sufficiently finely divided, and, in particular, not too much should be taken for bromination. Thus, 0·1 grm. of the rubber mentioned above was completely brominated by a single application of bromine water, while 0·5 grm. required four; there was no over-bromination on six applications. Commercial bromine contains sulphuric acid, so that with repeated brominations a figure may be obtained for the free sulphur in excess of the total sulphur. If the author's conditions are adhered to this is of small importance, since a similar quantity of bromine is used in the estimation of the free sulphur; and it is the difference between the figures, and not the actual figures, which is of importance. After a single bromination of 0·1 grm. substance, the vulcanisation sulphur, from the difference between free and total sulphur, was 2·06 per cent.; after four brominations, 2·61; and after six, 2·11. It is, therefore, not possible to assume that vulcanisation sulphur is oxidised out of rubber by bromine, and this is supported by the higher percentage of vulcanisation sulphur in hard rubber than is found in soft rubber. It is not surprising that Hinrichsen and Kindscher found a higher value for free sulphur from the sulphur in bromine water than from the sulphur dissolved in acetone. Budde (*ibid.*, 1909, **34**, 407) has already shown that acetone does not extract all the free sulphur. There is no material error due to loss of bromine from the bromide.

O. E. M.

Studies on Soil Humus. S. Davitt. (*J. Ind. and Eng. Chem.*, 1912, **4**, 601-604.) —Five samples of soil were experimented with, four being low-grade soils poor in humus from Tennessee, and one rich in humus from Florida. Two methods were employed for the removal of clay—firstly, that of Mooers and Hampton, involving extraction with 1 per cent. hydrochloric acid, followed by successive volumes of 4 per cent. ammonia. The extracts are repeatedly evaporated to dryness and baked. Some months of standing with water follow, to allow the clay to settle out. The second method employed was that of mechanical separation of the clay by long standing without evaporation to dryness. Some preliminary investigations are recorded dealing with the retention of humus from water solution by ferrous iron, ferric iron, and calcium chloride. After such treatment there always remained some organic matter in solution which could be removed more or less completely by a copper salt. A starch-like substance, which gave no iodine coloration, was present to the extent of from 4 to 10 per cent. in humus from Florida; it can be hydrolised by acids and also acted upon by diastase with subsequent acid hydrolysis. Both of these processes gave reducing sugars, one of which was obtained in a crystalline form, but could not be identified. The mother liquor from which this sugar was crystallised was optically inactive and easily fermentable. After fermentation it contained alcohol and acetic acid and still reduced alkaline copper solution.

H. F. E. H.

Study of the Acidity of Soils. A. Gregoire. (*Bull. Soc. Chim. Belg.*, 1912, **26**, 386-409.)—In a previous communication (ANALYST, 1912, 422) a method was described for the estimation of acidity based on the reaction between potassium iodide and iodate by the use of "Kjeldahl's solution." This reagent is capable of estimating the feeblest of organic acids, even those insoluble in water. At the same time it reacts also with inorganic compounds of acid function and shows marked acidity with aluminium compounds and silica. On applying the method to soils it was found that all soils, even those of alkaline reaction to litmus and containing notable proportions of calcium carbonate, showed some acidity with Kjeldahl's reagent, and it is necessary in interpreting the results to establish some limiting standard. Undoubtedly the main acidity of soils is due to the organic matter, and whilst the total acidity has little absolute significance, the ratio of Kjeldahl acidity to organic carbon is capable of giving very valuable information as to the normal or abnormal character of the soil. The results obtained by the author's method as a general rule run parallel with those shown by the litmus method of Larsen and Christensen, and the Kjeldahl reagent possesses the advantage of affording direct numerical expression of the acidity or hydrogen ions. In view of the close correlation between the acidity and the organic matter, the author has worked out a method for the estimation of the organic carbon by combustion of the soil previously treated with acid to decompose the carbonates. The combustion tube is of quartz, 20 to 22 mm. in diameter and 700 to 800 mm. long, connected with the usual supply of oxygen at one end and charged with a layer of about 100 mm. of copper oxide; about 5 grms. of soil are weighed out in one or two quartz boats. The arrangement for absorbing the carbon dioxide produced consists of a Pettenkofer tube, 18 to 20 mm. in diameter and 1·5 to 1·8 metre long. It has an inclination of 120 mm. per metre and is provided with three or four baffle cones of platinum to prevent the direct passage of the bubbles of gas. The absorption liquid is a solution of barium hydroxide. When the combustion is finished the further end of the absorption tube is attached to a filter-tube containing a plug of abestos, similar to, but larger than, those employed for collecting the cuprous oxide in the estimation of reducing sugars. The absorption tube is then detached from the furnace and placed in a vertical position. The filtration of the barium carbonate takes place fairly rapidly and the tube is rinsed down with boiled water. The barium carbonate in the filter tube is estimated gasometrically. Scheibler's apparatus is unsuitable because it has been found that the caoutchouc bag absorbs carbon dioxide. The carbon dioxide must therefore be generated in a glass vessel and measured by displacement of air, air only coming in contact with the rubber connections. The gas evolution vessel employed is shown in the accompanying sketch (see Fig.); it is a cylinder 45 mm. in diameter and 350 mm. long, surmounted by a stopcock and bulb which is charged with acid, whilst the lower end is ground for insertion either into the neck of a flask or into the filter-tube containing the barium carbonate. This vessel is adapted to lie in a large trough of water, whilst the upper end is connected by a rubber tube with the gas-measuring apparatus. Nitric acid of 1·20 sp. gr. is employed, since with hydrochloric acid the barium chloride is only sparingly soluble. A correction must be made for the solubility of the carbon dioxide in the acid liquid. The author points out that a fixed correction, such as is usually recommended, is quite inaccurate, since the solubility of the gas depends on its partial pressure, so that the correction varies with the relative quantity of gas evolved and air present. It is necessary, however, after evolution of the gas is complete, to close the stopcock and effect a thoroughly uniform mixture of the carbon dioxide and air remaining in the cylinder; the cock is then opened again, the levels readjusted and the reading taken. The correction for solubility is then found by means of the formula $\frac{S \times V \times H}{A \times 760}$, where S = the coeficient of solubility of pure carbon dioxide, V = the volume of carbon dioxide read off, H = the atmospheric pressure reduced to 0° C., and A = the volume of the evolution vessel, up to the stopcock minus the volume of the acid. The coefficients of solubility of pure carbon dioxide in 10 c.c. of nitric acid of 1·20 sp. gr. are given as follows, in terms of gas at 0° C. and 760 mm. pressure: At 10° C., 11·48 c.c.; 12° C., 11·28 c.c.; 14° C., 11·02 c.c.; 16° C.,



10·67 c.c.; 18° C., 10·28 c.c.; 20° C., 9·78 c.c.; 22° C., 9·25 c.c.; 24° C., 8·56 c.c. In the case of hydrochloric acid the coefficient of solubility may be taken as the same as in the case of water. J. F. B.

INORGANIC ANALYSIS.

Colour Reaction of Ammonia. P. Thomas. (*Bull. Soc. Chim.*, 1912, **11**, 796-799.)—The blue coloration which is developed when an ammonium compound is treated with a solution of phenol and a hypochlorite is as delicate as the reaction with Nessler's reagent, and, in the author's view, is more specific. It can be used in cases where Nessler's test fails, as, for instance, in the case of liquids containing proteins, more or less cloudy. In carrying out the quantitative test a solution of ammonium chloride containing 1 grm. in 10 litres is employed as a standard; increasing quantities of this are placed in a series of glasses, the volume being made up to 5 c.c. in each case. Then are added 1 c.c. of a 4 per cent. solution of phenol and 1 c.c. of "eau de Javel," the commercial liquor diluted to 10 per cent. The blue coloration develops gradually after mixing and is visible at a dilution of 1 in 500,000. The reaction is applicable to the estimation of ammonia in water, but so far it has not been possible to use it in the case of organic secretions. In testing various nitrogenous substances in order to ascertain how far this reaction is peculiar to ammonium compounds, it is difficult to be quite sure of the absence of ammonia as an impurity. Among the amino-acids glycocoll would appear to be the only one which shows the coloration with any intensity; certain others develop a slight colour, but only at concentrations above 1 per cent. Acetamide shows no colour, but urea gives a pale green. The amines of the fatty series are not readily obtainable perfectly free from ammonia. It would seem that all the primary amines give the same coloration to a greater or less extent, methylamine and ethylamine react with great intensity. The secondary amines react feebly, but their purity is doubtful; the tertiary amines do not give the reaction. J. F. B.

Electro-analytical Estimation of Copper in Pyrites. W. D. Treadwell. (*Chem. Zeit.*, 1912, **36**, 961.)—Copper may be deposited quantitatively by electrolysis of a solution of pyrites in aqua regia without eliminating the aqua regia. If the following directions be adhered to, the deposit will never contain more than 1 mgrm. of iron and lead, but since it invariably includes some iron, it should always be redissolved in nitric acid and redeposited. The solution of 3 to 4 grms. pyrites, which need not be filtered from gangue, is diluted to 100 to 150 c.c., and ammonia is added until a slight permanent turbidity is produced. About 10 grms. of Rochelle salt (about 8 grms. per grm. of iron present) is next added to hold up the iron. Ammonium sulphate (5 grms.) and ammonia (20 c.c.) are added, and the solution is electrolysed in the cold, using a gauze cathode which is rotated at a moderate speed, and preferably a cylindrical anode. The cathode must remain covered by the liquid or copper will redissolve from it under the influence of oxygen and ammonia. With a difference of potential between the terminals of 2 volts, corresponding to a current varying from about 2·7 to 0·7 amp., 0·5 grm. copper may be deposited in sixty to seventy-five minutes from a solution containing 1·5 grms. iron. If speed is no object, the use of a rotating cathode may be avoided, and the terminals of the apparatus connected to those of a single lead accumulator and left overnight. In this case the action is started with the temperature of the electrolyte at about 40° C., and not more pyrites than corresponds to 0·3 grm. copper should be taken for the estimation. In any case, the deposit is washed with a jet of water as the cathode is lifted out of the bath, and is redissolved in 5 c.c. of nitric acid, and the solution is boiled to expel nitrous gases and re-electrolysed, preferably with the use of a rapidly rotating cathode, and the current from two Edison accumulators short circuited through the bath (about 2·6 volts). This second electrolysis occupies only ten to fifteen minutes. The first one may be accelerated by the use of hydrazine sulphate or sodium bisulphite to reduce the iron to the ferrous state, instead of leaving this work to the current, but this is not permissible if the pyrites contains arsenic, for these reagents reduce arsenic to the arsenious condition, with the result that arsenic is deposited on the cathode with the copper. G. C. J.

Reaction of Gold Salts with *m*-Phenylenediamine. J. A. Siemssen. (*Chem. Zeit.*, 1912, **36**, 934.)—A solution of *m*-phenylenediamine (5 : 1,000) is a sensitive reagent for gold salts, and will give an immediate violet coloration with a 0·005 per cent. solution of gold chloride. With stronger solutions of the gold salt (0·5 per cent.) the reagent gives colorations ranging from yellow to dark brown. The *m*-phenylenediamine solution must be decolorised, if necessary, by boiling with freshly ignited animal charcoal. C. A. M.

Separation of Iron from Titanium. F. Bourion. (*Ann. Chim. anal.*, 1912, **17**, 281-284.)—The extreme error of the method, which has been described before (ANALYST, 1912, 323), is ± 2 mgrms. when operating on 0·5 grm. or less of the mixed oxides. G. C. J.

Quantitative Separation of Lanthanum from Yttrium. C. James and T. O. Smith. (*Chem. News*, 1912, **106**, 73-74.)—The following method was found to give a more perfect separation than the potassium sulphate method. It depends on the insolubility of lanthanum magnesium nitrate in concentrated nitric acid saturated with bismuth magnesium nitrate. Yttrium forms no double nitrate with magnesium. The solution containing lanthanum and yttrium as nitrates is evaporated to dryness, and the residue is treated with 25 c.c. of concentrated nitric acid saturated with bismuth magnesium nitrate. Solid bismuth magnesium nitrate (10 grms.) and 1 grm. of magnesium nitrate are added, and the mixture warmed and stirred, and then allowed to stand for twenty-four hours. The crystalline deposit is filtered off on a Gooch crucible, and washed with nitric acid saturated with bismuth magnesium nitrate. The filtrates and washing are evaporated to dryness, the residue is treated with 200 c.c. of cold water, and bismuth is precipitated with hydrogen sulphide. The bismuth sulphide is filtered off and washed with cold water, and the filtrate is boiled to expel hydrogen sulphide, cooled, treated with ammonium chloride, and nearly neutralised with ammonia. Yttrium is then precipitated by means of oxalic acid, and the oxalate ignited to the oxide and weighed. The lanthanum magnesium nitrate and bismuth magnesium nitrate in the Gooch crucible are washed into a beaker, bismuth is precipitated as sulphide, and in the filtrate from the bismuth sulphide lanthanum is precipitated in a manner exactly similar to that followed in the case of yttrium. With 0·2 grm. of lanthana and 0·2 grm. of yttria present, the amount of each earth may be determined with an error which does not exceed ± 4 mgrms. It is pointed out that the error would probably be greater if the method were applied to the separation of lanthanum from holmium and dysprosium, since these elements have a tendency—slight though it may be—to form double nitrates with magnesium. On the other hand, the method might be expected to give a nearly perfect separation of lanthanum from those members of the yttrium group, the nitrates of which are more soluble than that of yttrium itself.

In the author's hands the potassium sulphate method underestimates yttrium by more than 10 per cent., and no satisfactory results were obtained by substituting sodium sulphate for potassium sulphate, nor by methods depending on the different solubilities of the formates and cacodylates of lanthanum and yttrium. G. C. J.

Determination of Lithium. L. R. Milford. (*J. Ind. and Eng. Chem.*, 1912, 4, 595-597.)—Of mineral waters containing much sodium and potassium relatively to lithium, 250 c.c. are evaporated to dryness in a large platinum dish, with occasional addition of a few drops of hydrochloric acid. The residue is heated to 120° C. for half an hour to render silica insoluble, and is then moistened with hydrochloric acid and rubbed up with 25 c.c. of 95 per cent. alcohol. The alcoholic extract is filtered, and the insoluble matter washed with 95 per cent. alcohol so long as lithium can be detected spectroscopically. The alcoholic filtrate and washings are evaporated to dryness, a little hydrochloric acid being added towards the end, and the residue is extracted and the extract filtered as before, but using absolute alcohol in place of weaker spirit. The alcoholic filtrate is evaporated to dryness, and the residue taken up in a little dilute hydrochloric acid. Magnesia is precipitated by addition of an excess of calcium hydroxide, and the solution is filtered, and most of the lime precipitated by addition of ammonia and ammonium oxalate. After standing overnight, the calcium oxalate is filtered off and washed with at least 100 c.c. of water, or until lithium can no longer be detected. The filtrate and washings are evaporated to dryness, ammonium salts expelled at a low temperature, and the residue taken up once more in a little hydrochloric acid. Ammonia and ammonium oxalate are added again, any precipitate which separates at once or on concentrating is filtered off, and the filtrate is evaporated to dryness, and ammonium salts expelled by heating in platinum just short of redness. The residue is taken up in water, filtered and washed, using as little wash-water as possible, and is then evaporated with hydrochloric acid and chlorine-water to decompose any bromide or iodide. The ignited residue is taken up with a little water, and the solution, acidified by a few drops of hydrochloric acid, is transferred to a large platinum dish. About 30 c.c. of amyl alcohol are added and the water boiled off, with constant stirring to prevent bumping. A drop of dilute hydrochloric acid is added to reconvert traces of lithium hydrate to chloride, and the solution is concentrated to about 15 c.c. on the steam-bath, and then filtered into a platinum dish and evaporated to dryness. This treatment with amyl alcohol separates most of the sodium and potassium chloride which escape separation by means of alcohol in the earlier stages of the process. The residue of lithium chloride is treated with water and a few drops of sulphuric acid and evaporated to dryness, the excess of acid is expelled by heating to dull redness, and the residue again taken up in water. Ammonia is added to precipitate the last trace of magnesia, which is filtered off and washed with boiling water. The filtrate and washings are evaporated to dryness, freed from ammonium salts, and the treatment with ammonia repeated until no further precipitate of magnesia is formed. The lithium is finally weighed as sulphate, and a deduction of 1·7 mgrms. is made to correct for the solubility of sodium and potassium chlorides in amyl alcohol.

G. C. J.

Gravimetric Estimation of Magnesium. L. Karaoglanow. (*Jahrbuch d. Univ. Sofia,* 1910-1911, 1-57; through *Chem. Zentralb.*, 1912, II., 548-549.)—Magnesium ammonium phosphate, precipitated from solutions of magnesium chloride or nitrate, should be washed with ammonia containing ammonium salts, but precipitates obtained from the sulphate are best washed with 2·5 per cent. ammonia. The method of Neubauer always gives higher results than the method of Gibbs or that of Schmitz, and the results obtained by the latter methods agree closely with those yielded by the oxide and sulphate methods, which the author has slightly modified with a view to making them sufficiently accurate to serve as control methods in his experiments on the phosphate method. The high results yielded by Neubauer's method are not due to the formation of $(NH_4)_4Mg(PO_4)_2$, as Neubauer, and also Gooch and Austin, have assumed, but are probably due to the power of adsorption possessed by the amorphous or granular magnesium ammonium phosphate. Pellet's method is useless for quantitative purposes, since no definite magnesium sulphopyrophosphate is obtained, but only an equilibrium between magnesium pyrophosphate and sulphuric acid.

G. C. J.

Volumetric Estimation of Manganese. L. Karaoglanow. (*Jahrb. der Univ. Sofia,* 1910-1911, 1-33; through *Chem. Zentralbl.*, 1912, II., 548.)—Some of the disadvantages of Volhard's method disappear when it is applied to solutions of manganous nitrate instead of to solutions of the sulphate. For example, the titration can be carried out in presence of ferric nitrate and aluminium nitrate, whereas the corresponding sulphates hinder the action of permanganate on manganous sulphate. Moreover, manganous nitrate can be titrated in nitric acid solution, whereas in sulphuric acid solution manganion is not oxidised by permanganate. The presence of the nitrates of barium, zinc, potassium, and silver, further the reaction between manganous nitrate and permanganate. Ferric nitrate and aluminium nitrate in high concentration limit it, their action depending on their tendency to hydrolytic dissociation in aqueous solution, the nitric acid thus set free being the agent which interferes with the oxidation. The method always gives results somewhat below the truth, the most accurate results being those obtained in presence of some ferric nitrate. The most convenient form of the method, however, is that in which silver nitrate is added to the solution to be titrated, the results render such circumstances being "sufficiently exact." Silver nitrate reacts with manganous acid to form silver manganite, the amount of the latter formed depending on the relative concentrations of silver nitrate, manganous nitrate, and manganous acid. In practice, the precipitate consists mainly of manganous acid, the greater part of the silver nitrate remaining unchanged in the solution. If other nitrates are present, even less silver manganite is formed, the other metals replacing the silver of silver manganite. These other metallic ions exercise a similar influence on the divalent manganese of manganous manganite. This is the explanation of the favourable influence of metallic salts on the reaction between permanganate and manganous nitrate. Equivalent quantities of different metallic nitrates displace different quantities of silver or manganese from their combination with manganous acid; the order is ferric, silver, zinc, aluminium, barium, and potassium nitrate. The same order applies to the accuracy of the results when manganese nitrate is titrated with permanganate, the most exact results being those obtained in presence of a reasonable amount of ferric nitrate. The variation in the results cannot be ascribed to disturbed equilibrium between manganous manganite and metallic nitrate, but depends on the fact that the formation of manganous manganite cannot wholly be prevented even if the titration be conducted in presence of a great excess of another salt. Once manganous manganite is formed, however, it is with difficulty either oxidised by permanganate or substituted by another metal, since the system is heterogeneous. The author's work proves the untrustworthiness of Rössler's method for the volumetric estimation of manganese, and throws doubt on Rose's method for the preparation of silver manganite, which afforded the basis of Rössler's method. G. C. J.

Absorption of Ultra-violet Rays in Ozone, and its Application to the Estimation of Ozone in Small Concentrations. F. Krueger and M. Moeller. (*Physikal. Zeitsch.*, 1912, **13**, 729; through *Chem. Zentralbl.*, 1912, II., 748.)—The rapid absorption in the region of wave-lengths from 200 to 300 $\mu\mu$ may be used for the estimation of ozone if the coefficients of absorption for the several wave-lengths are accurately known. The authors find that the maximum absorption has the position assigned to it by E. Meyer (*Annalen der Physik.*, 1903, **12**, 849), but that his coefficients of absorption require correction. The light from a quartz mercury vapour lamp was passed through a quartz prism, a slit, the absorption-tube, and a photo-electric potassium cell. The current, measured by means of a quadrant electrometer, is a measure of the intensity of the light. Comparative tests with known concentrations of ozone gave satisfactory results with the new data. The optical method is more sensitive than the chemical, and can be carried out on lower concentrations. O. E. M.

Estimation of Inorganic Phosphorus in Plant Substances. R. C. Collison. (*J. Ind. and Eng. Chem.*, 1912, **4**, 606-609.)—Dilute acid extracts of plant substances contain proteins, carbohydrates, and, in the case of cereals, salts of phytic acid, besides inorganic salts. All these substances, especially the salts of phytic acid, inhibit the formation of ammonium phosphomolybdate. The author previously (*Ohio Agr. Exp.*

Sta. Bull., No. 215) recommended a preliminary extraction with 0·2 per cent. hydrochloric acid, followed by precipitation with magnesia mixture. The dried precipitate was then thoroughly extracted with a measured volume of acid alcohol, the mixture filtered, and phosphorus determined in an aliquot portion of the filtrate, thus avoiding the precipitation of phosphorus in presence of proteins and salts of phytic acid. The author has since found that cold alcohol (94 per cent.) containing hydrochloric or nitric acid (0·2 per cent.) dissolves the common phosphates which are insoluble in neutral or slightly alkaline alcohol. Proteins in general, nucleinic acid from yeast, carbohydrates and salts of phytic acid, are mostly insoluble. He therefore recommends such an acid alcohol extraction of the material followed by filtration, thus leaving behind the undissolved organic phosphorous in the residue. The method was fully investigated on soya beans, rice polish (on account of its high content of phytin), and maize germ meal. Known amounts of inorganic phosphorus were added to these materials and recovered with accuracy. Ten grms. of the finely-ground material is placed in a 400 c.c. flask and covered with 300 c.c. of the acid alcohol solution referred to, the whole being shaken every five minutes for three hours. After filtration an aliquot portion is made just alkaline, with ammonia, and then allowed to stand eight to twelve hours. The precipitate is filtered off, washed with 94 per cent. alcohol made faintly ammoniacal, and then dried, after which it is dissolved in 100 c.c. of 0·5 per cent. nitric acid. The material is again filtered and precipitated with acid molybdate solution. A further advantage of the proposed method is that alcohol extraction tends to prevent enzymic and bacterial decomposition of organic phosphorus compounds, evidence being given that in many cases variations in results have been due to the splitting off of inorganic phosphorus from organic compounds. Analyses of some thirteen substances are recorded by the acid water and acid alcohol methods, with and without the addition of known amounts of inorganic phosphorus, in all cases the results agreeing with the theoretical figures when the latter method is used, and in five cases only when the acid water method was adopted, these five being materials which do not give much organic matter on extraction. H. F. E. H.

New Compensation Method for the Comparison of Quantities of Radium, and some Applications of this Method. E. Rutherford and G. Chadwick.

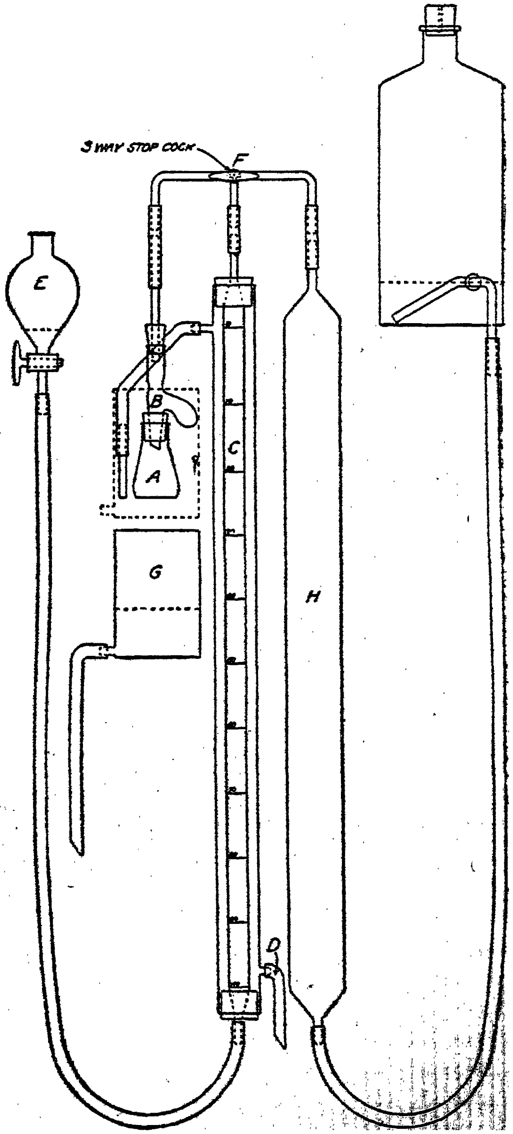
(*Le Radium*, 1912, **9**, 195; through *Chem. Zentralbl.*, 1912, II., 752.)—A preparation of unknown radium content is best compared with a standard by means of the γ-radiation. It is a necessary condition that the preparation shall contain no mesothorium or radiothorium, as both give off γ-rays nearly equal in effect to those of radium. The intensities should not be greatly different, as with ratios of 10 to 1, for example, it is difficult to be certain that there is complete saturation of the ionisation current in the electroscope. To avoid this difficulty, the authors have evolved a compensation method, in which the γ-rays from the radium preparation produce a saturation current, which is compensated in an ionisation vessel by an opposed constant ionisation current from a preparation of uranium oxide. Exact compensation is effected, with the help of a Dolezalek electrometer, by adjusting the distance from the ionisation vessel of the radium preparations, the intensity of which is then proportional to the square of this distance. Corrections for the absorption in air, and, if necessary, in glass, are applied in accurate determinations. Further corrections, for which the original should be consulted, are necessary for the estimation of radium emanations. The γ-activity of a considerable quantity of radium emanation gave by this method an exponential decrease, with a limit of 3,854 days; a tube filled with radium emanation reaches its maximum in 255 minutes. The emission of γ-rays was not affected by bringing the emanation into a powerful magnetic field. O. E. M.

Continuous Fractional Distillation of Water. W. R. Bousfield. (*J. Chem. Soc.*, 1912, **101**, 1443-1453.)—Water having a conductivity represented by $K_{18} = 1 \times 10^{-6}$ is taken as having unit conductivity. In a previous paper (*J. Chem. Soc.*, 1905, **87**, 740) an apparatus yielding water of this order of purity by continuous fractional distillation of tap water was dealt with by the author, and he now describes, with drawings and full details (for which reference must be made to the original), two forms of still more readily managed, and of a simpler type. The first of these—intended for everyday purposes—yields about 30 litres of water per twenty-four hours, two-thirds of which has a conductivity of about 0·8, the rest being of ordinary character. If a strong solution of alkaline potassium permanganate is supplied with the feed-water at the rate of about 10 drops a minute, water of a slightly lower conductivity may be continuously produced. The other apparatus is suited for the continuous redistillation of water under diminished pressure, and by its use water having a conductivity of only 0·5 may be continuously produced by the redistillation of the ordinary product of the first still. Criticising Hartley, Campbell, and Poole (*J. Chem. Soc.*, 1908, **93**, 428), it is shown by several experiments that marked differences characterise the conductivities of the condensation products when waters containing traces of ammonia and acetic acid respectively are distilled and allowed to condense on (1) warm and (2) cold surfaces. In the distillation of ordinary water the "hot-surface" product is the purer when employing the first of the stills figured in the paper; while in the second, as in the still orginally described (*loc. cit.*), the cold-surface product is the purer, and is probably of a higher degree of purity than any hitherto produced on a large scale by any previous arrangement of still. The purest product is obtained from the hot surface, when the rate of distillation is so arranged that the proportions of distillate collected from the hot and cold surfaces are about 2 to 1. Working so as to obtain this ratio, with a total output of 1½ litres per hour, with 3 inches gas pressure, the following conductivities were obtained from the first still:

	K_{18}.	
	Hot-Surface Product.	Cold-Surface Product.
1. From tap water only	0·90	10·0
2. With a large addition of alkaline permanganate	1·20	2·0
3. With 10 drops per minute ...	0·75	2·8

The paper closes with a description and illustration of the second, or "three-bottle," still in series, where an electric heating source is employed in the first bottle, the "hot-surface" product collecting in the second bottle, and the "cold-surface" water going into the third. H. F. E. H.

Calcium Carbide Method for Estimation of Moisture. H. C. McNeil. (*Chem. Eng.*, 1912, **16**, 38-40.)—This method has been made use of for a variety of analyses by different observers (*cf.* P. V. Dupré, ANALYST, 1909, **30**, 266; 1906, **31**, 213; R. A. Cripps and J. A. Brown, 1909, **34**, 519; I. Masson, 1911, **36**, 112). The author's apparatus for measuring the acetylene given off by reaction with the moisture-containing substance, shown in the figure, has been used for the examination of soaps, paints, leather powder, and infants' foods.



The reaction flask, *A*, has a capacity of 25 c.c., and into it a weighed amount of the sample in powder form is introduced. Barium sulphate, silica or similar material is used to mix with the substance, if necessary, so that the carbide will readily mix with it; or the sample may be dissolved or suspended in a liquid, such as toluene, amyl alcohol, or even kerosene and heavy mineral oils. The tube *B* is charged by filling the side projection with carbide ground to a fine powder, a cotton-wool plug pushed down to the constriction, and a little more carbide, covered with wool, placed alternately until the tube is loosely packed, to prevent loss of moisture. The stopcock, *F*, is opened to the air by pulling it partly out, after connecting *A* with *B*, and *B* with *C* by means of tubing, etc., as shown. The flask *A* and tube *B* are surrounded with the vessel *G*, and water is run through *C* and *G* till the apparatus is at the temperature of the water, and when the burette is reading zero by adjustment with *E* the tap *F* is turned so that *B* and *C* are in communication. A little carbide is then tipped into *A* and the rate of reaction carefully controlled. The flask is thoroughly shaken, and the mixture heated for about fifteen minutes, by surrounding the vessel with a bath of boiling water. The apparatus is then reduced to the initial temperature, and the measurements made. The tube *H* is an auxiliary container for use in the case of substances containing much moisture, or of such a nature that a fair sample in small amounts cannot be obtained. In such cases *F* is turned to connect *C*, *H*, and *A*, and the resultant gas is lead from *H* into *C* and cooled, measured, and passed out in portions. Mercury is used in the apparatus, or strong brine saturated with acetylene, and coloured with alkali and phenolphthaleïn.

The apparatus should be standardised, so that the amount of gas given off from carbide by a definite amount of water is known. Practically, 1 grm. of water yields about 582 c.c.

Paints, pastes, and soaps, should be mixed with freshly ignited barium sulphate in sufficient amount to overcome pastiness, and then sand is added to make the mass granular. Alcohol may be used in the case of soaps, instead of the dry method just described, provided that it is first treated with calcium carbide and saturated with acetylene. The infants' foods examined reacted only slowly, the reaction being in some cases incomplete after one hour at 100° C. A. R. T.

Assay of Sodium Peroxide for Bleaching Purposes. W. Herbig. (*Färber Zeit.*, 1912, **23**, 193-196; through *Chem. Zentralbl.*, 1912, II., 636-637.)—In the author's experience the gasometric method for the assay of sodium peroxide often gives too high results, values sometimes equivalent to 114 per cent. of sodium peroxide, which can only be explained by the presence of some higher oxide—*e.g.*, the tri- or tetroxide of sodium. Such higher oxides are of no interest to the bleacher. With the permanganate titration method satisfactory results are obtained by the following procedure: A mixture of 200 c.c. of water and 200 c.c. of dilute sulphuric acid (1 : 5) is cooled to 0° C., by the addition of some clean ice, the permanganate-consumption of which has been previously determined. The height of the liquid in the beaker should be 10 or 12 cm. Then a weighing tube containing 0·2 to 0·3 grm. of sodium peroxide is quickly dropped in a horizontal position to the bottom of the beaker. The air in the weighing tube only allows the acid to come gradually in contact with the peroxide, so that decomposition is slow. If the acid attacks the peroxide all at once the results show considerable variations. The iodometric method may be carried out in a similar manner. The peroxide enclosed in a weighing tube is introduced into a 10 per cent. solution of potassium iodide acidified with hydrochloric acid, and the flask is kept stoppered for forty-five minutes; the liquid is then diluted largely, and the iodine titrated with thiosulphate. The results are concordant if the iodide solution be sufficiently strong during the reaction, but it must be sufficiently diluted before the titration; the acid must be present in excess, and, above all, the thiosulphate solution must only be added drop by drop. Certain samples of sodium peroxide contain greyish black grains consisting apparently of sodium ferrate; such impurities are liable to be very injurious to the cloth in the bleaching process. J. F. B.

Volumetric Determination of Tin. W. W. Patrick and G. C. Wilsnack. (*J. Ind. and Eng. Chem.*, 1912, **4**, 597-599.)—If the alloy does not contain more than 5 per cent. of lead or copper, an amount corresponding to 0·5 grm. or less of tin is taken for analysis, and if the sample contains less than 0·15 grm. of antimony, some powdered antimony is weighed out with it. Solution is effected in a 500 c.c. Erlenmeyer flask by heating with 10 c.c. of sulphuric acid. To the cooled solution 10 c.c. of water is added, and this is followed by 60 c.c. of hydrochloric acid, and a further 40 c.c. of water. The flask is fitted with a four-hole rubber stopper. Through one hole passes a tube for the introduction of carbon dioxide to the bottom of the flask. Another hole carries a short bend which is connected by flexible tubing to a 100 c.c. pipette which dips into water, and serves to prevent air being sucked into the apparatus if the flow of carbon dioxide is not quick enough to prevent the formation of a partial vacuum when the contents of the flask are cooled. A third hole carries a glass rod of such diameter that it can easily be moved up and down. In its lowest position it should nearly touch the bottom of the flask, and at its lower end it has sealed to it a platinum hook to which is attached a spiral of very pure iron made from a 4-inch by ⅛-inch strip of ⅛-inch plate. The fourth hole carries a piece of ½-inch tubing, drawn out to a jet below and with a lateral hole blown on it just above the jet. This tube serves for the introduction of the iodine solution used for titrating the tin, and also for the introduction of water to wash the iron coil when this is raised out of the liquid. With this latter object the jet is bent at a slight angle from the vertical. The lateral hole allows carbon dioxide to escape without impeding the even delivery of iodine solution drop by drop. This stopper being fitted to the flask, and the jet tube being suitably plugged, the air in the flask is displaced by carbon dioxide, and the contents of the flask are maintained in gentle ebullition for twenty-five minutes, and then cooled in running water, the current of carbon dioxide being continued all the time. The iron wire is raised out of the liquid and washed free from tin solution by means of freshly boiled water admitted through the jet tube. The tin is then titrated with iodine and starch, after raising the iron coil to such a position that iodine falling from the jet cannot come in contact with it.

Alloys containing more than 5 per cent. of copper or lead are decomposed by covering with 10 c.c. of concentrated nitric acid, followed by evaporation to dryness. The residue is baked strongly for fifteen minutes, and, when cool again, is rubbed up with 20 c.c. of dilute (1 : 1) nitric acid. The insoluble matter is filtered off, washed with hot water, and then transferred with the filter-paper to a 500 c.c. Erlenmeyer flask, in which it is digested, at first at a gentle heat, with 20 c.c. of sulphuric acid and 2 grm. of potassium sulphate. Before the liquid loses its black colour, it is allowed to cool, and any carbon on the sides of the flask washed down with water, aided, if necessary, by a "policeman." The digestion is then continued until the solution is colourless. To the cooled solution, 50 c.c. of water and 45 c.c. of hydrochloric acid are added, and the reduction and titration of the tin completed in the manner already described.

The iodine solution is standardised on the purest tin obtainable. The maximum error of the method is 1 in 400 if sufficient of the alloy is taken to give a large reading on the iodine burette. G. C. J.

Analysis of the Waters of the Thermal Springs of Bath. I. Masson and W. Ramsay. (*J. Chem. Soc.*, 1912, **101**, 1370-1376.)—The King's spring, the largest of the three main springs, delivers half a million gallons in twenty-four hours at a temperature of 50° C. Potash drinking glasses, after prolonged use, turn yellow owing to the niton or radium emanation contained in the water having liberated potassium in an atomic state of subdivision. Soda glass, similarly exposed, turns pale violet. The water has a density of 1·0175 at 4° C., and a freezing-point of $-0·07°$ C., and gas escapes at the rate of 4,927 litres in twenty-four hours. Oxygen, hydrogen, and methane were absent from this gas, and 168 c.c. of residual inactive gases freed from CO_2 by potash and from nitrogen by passing through red-hot tubes charged with magnesium lime, were fractionally condensed in charcoal cooled with liquid air. Portion 1 measured 96 c.c., and weighed 1·706 grm. per litre. Helium and krypton were absent, and calculation showed the presence of 88·7 c.c. of argon and 7·9 c.c. neon. Portion 2 measured 61 c.c., weighed 1·3509 grm. per litre, and consisted of 31·1 c.c. of argon and 29·9 c.c. neon. Helium lines were absent. Portion 3 was uncondensable by charcoal, the weight of a litre was 0·2681 grm., and the fraction measured 5·6 c.c.; 4·9 c.c. of helium and 0·7 c.c. of neon were calculated to be present. Adding these three portions together, the total gas contained per litre is CO_2, 36 c.c.; nitrogen, 954·1 c.c.; argon, 7·263 c.c.; neon, 2·334 c.c.; and helium, 0·297 c.c. Bath gas, therefore, contains 0·779 times as much argon, 188·1 times as much neon, and 72·8 times as much helium as atmospheric air. In the total of 5,363 litres of gas escaping daily from the three springs, there are 39 litres of argon, 12·5 litres of neon, and 1·6 litres of helium. Niton or radium emanation is also present in too small quantity for chemical estimation, but was measured by comparing the rate of discharge of an electroscope filled with Bath gas, with the effect produced by the niton accumulated during a definite time from a standard solution of radium bromide containing $\frac{1}{10000}$ mgrm. of metallic radium. The niton in a million litres of gas from the King's spring was thus found to be in equilibrium with 33·65 mgrms. of radium. The residue left on evaporating the water had the following composition in mgrms. per litre: Li, 0·16; Na, 181·1; K, 16·7; Mg, 4·1; Ca, 335·7; Sr, 3·2; Fe, 40·2; Al, 9·7; As, trace; Cl, 154·8; Br, 4·4; SO_4, 1054; CO_3, 15·8; SiO_3—. Total, 1811·0. The rare earths were absent. Radium, as metal, amounted to 0·1387 mgrms. in a million litres, the small quantity being due to the high percentage of sulphate. Some deposit on the old Roman lead pipes still at the spring consisted mainly of calcium sulphates. The radium in it was determined, since in 1,760 years half of that originally present would have changed into niton and its products. One part of radium in 10 million parts by weight of the deposit were found. Radium was not detectable in the modern deposit. H. F. E. H.

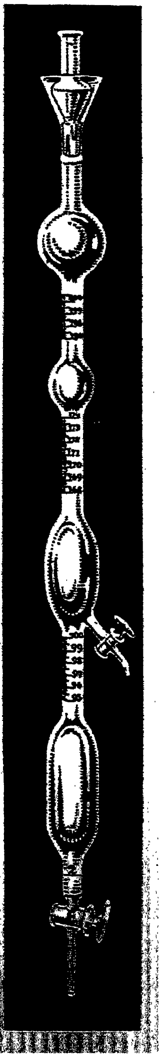
APPARATUS, ETC.

Application of Light Energy to the Study of Certain Problems in Analytical Chemistry. M. Landau. (*Compt. rend.*, 1912, **155**, 403-406.)—The author has studied the action of ultra-violet rays on certain hydrocarbons and carbon monoxide. This action is of two kinds: It causes the polymerisation of unsaturated gaseous hydrocarbons into liquid and solid bodies, whilst in presence of excess of oxygen it brings about photocombustion of hydrocarbon and carbon monoxide gases. The polymerising action is very slow, even when a powerful quartz mercury lamp of 500 volts is used at a distance of 4 cm. In the case of ethylene, the shrinkage of a volume of the gas amounted to 71 per cent. after nineteen hours, whilst a much longer exposure was required to produce complete condensation. In the case of acetylene the action was still slower. The photocombustion was studied in the case of ethane mixed with an excess of oxygen; combustion was complete after sixteen hours' exposure. The combustion of methane was more difficult, and incomplete in the time devoted to its study. That of carbon monoxide amounted to 72 per cent. in fifteen and a half hours, and was complete after longer exposure. In the combustion of hydrocarbons, intermediate products, including carbon monoxide, were formed. The combustions were carried out at a temperature of 80° C. The above reactions were applied to the analysis of a mixture of ethane, ethylene, and hydrogen. Such a mixture cannot be resolved by explosive combustion, because ethylene and hydrogen together are equivalent to ethane; but by a very prolonged exposure to the ultra-violet rays in absence of oxygen the ethylene may be condensed by photo-polymerisation and the contraction measured; oxygen is then added in excess, and the residue submitted to photocombustion. The spectra of the gaseous hydrocarbons show a strong absorption of ultra-violet rays whereas those of carbon monoxide and dioxide do not.

J. F. B.

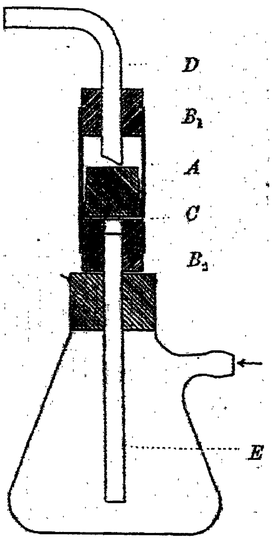
The "Sapometer." C. Huggenberg and H. Stadlinger. (*Chem. Zeit.*, 1912, **36**, 938.)—The special form of burette here illustrated is recommended for use in soap analysis. A weighed quantity (about 10 grms.) of the soap is dissolved in warm water, the solution poured into the "sapometer," which has previously been charged with 30 c.c. of $\frac{N}{1}$ sulphuric acid, and warm water added to bring the total contents to about 97 to 100 on the bottom scale. After the mixture is cold the fatty acids are extracted by shaking with ether or a mixture of ether and petroleum spirit, which is added until the contents reach a mark on the top scale. Any aliquot part of the upper ethereal solution may then be drawn off through the tap at the side and evaporated, while the lower layer is used for other estimations. The apparatus may be obtained from Altmann, Luisenstrasse, 47, Berlin.

C. A. M.



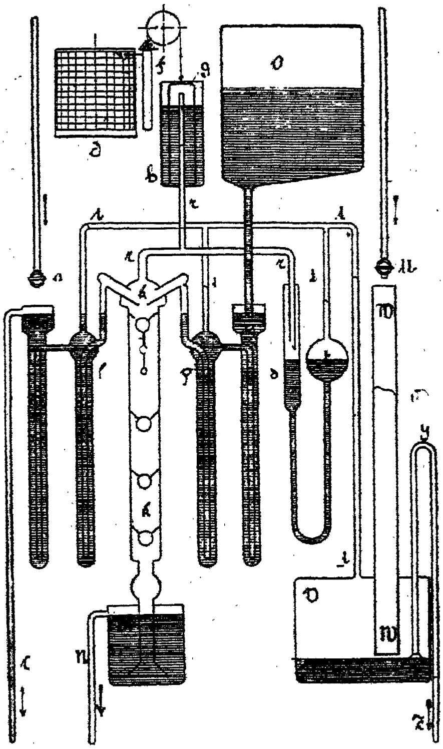
Apparatus for Automatically Decanting and Washing Precipitates. E. Sinkinson. (*Chem. News*, 1912, **106**, 49-50.)—The decanting and washing parts of the apparatus are separate, the former consisting of supports for the funnel and the beaker, containing the precipitate and liquid, and the latter of a rotating jet worked electrically.

The funnel containing the filter-paper is supported in a ring at one end of a balance-arm, and is counterpoised by a sliding weight on the other arm. By moving the weight the amount of liquid run into the filter can be regulated. When sufficient liquid has run into the filter, the funnel drops and is arrested by a fixed support, which carries four mercury cups serving to make and break electrical contact, and so controlling the motors regulating the supply of liquid to the filter. The beaker containing the liquid is supported on a sloping table, the slope being controlled by a motor. Thus, when the funnel drops, the motor is reversed and the liquid ceases to pass into the filter. When decantation is complete, and the precipitate has been transferred to the filter, the washing attachment is brought into use, the jet of wash-water being rotated by means of a worm-gearing electrically actuated. The operations of decantation and washing are carried out automatically, and the apparatus requires no attention except when the washing attachment is to be connected. The original paper is illustrated by four diagrams of the apparatus. A. R. T.

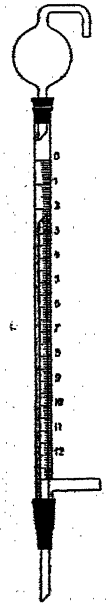


Valve for Filter Pumps. H. B. Hutchinson. (*Chem. News*, 1912, **106**, 99.)—This valve, which is of special use when the water supply is subject to much variation, consists of a stout glass-tube, *A*, $\frac{1}{2}$ inch to $\frac{3}{4}$ inch diameter fitted with two single-bored rubber corks, B_1 and B_2, between which is placed a solid rubber bung, *C*, with its wider end downwards, and fitting loosely into the tube. A glass tube, *D*, with an oblique lower end, is pushed through B_1, until it is about 1 to 2 mm. from the upper surface of the bung, *C*, and a second tube, *E*, is brought sufficiently far through B_2 to enable it to act as a support without bulging the upper surface of the cork. The valve may be used alone or attached to a small filter flask as shown. The latter way keeps the corks moist, which is an advantage, but when the valve is intended only as an air valve, it is best moistened with dilute glycerol. The tube *D* is then attached to the pump, and *E* on the side-tube of the filter-flask is joined to the filtering or vacuum distillation apparatus. Messrs. Gallenkamp supply the valve. H. F. E. H.

Apparatus for Automatic Analysis of Liquids. (*Chem. Zeit.*, 1912, **36**, 962.)—The apparatus figured serves for the automatic and periodical estimation of any constituent of a liquid which yields a gas on admixture with an appropriate liquid reagent—for example, for the estimation of ammonia in aqueous solution by the hypobromite reaction. The reagent is stored in the vessel *o* whilst the liquid to be controlled flows steadily through the cock *a* into *b*, continually overflowing through *c*. The whole apparatus is operated by the syphon tank *v*, and the



periodicity of its operation is controlled by the cock *u*, connected to a water-supply system. As the tank and tube *w* fill, the air-pressure forces the liquid in the bulb *t* into *s*, thus sealing the end of the tube *r*, which is the sole route by which gas can escape from the gas-bell *g*. With increasing head of water in *w* the (ammoniacal) liquid in the measuring tube *l* is forced into the reaction chamber *k*, and at the same time a measured portion of the reagent (hypobromite solution) is forced from *p* into *k*. The reaction chamber *k* is provided with means to ensure intimate mixture of the reacting liquids and with an automatic overflow. The gas generated in *k* passes through *r* to the gas-bell *g*, which rises, and in rising lowers a pencil which traces a continuous line on the clockwork drum *d*. Finally, the syphon *y* operates, the tank empties itself, the level of the liquid in *s* falls sufficiently to allow the gas in the bell to escape through *r*, the reagent measure *p* refills immediately, and the measure *l* fills more slowly with the liquid to be tested. The cycle of operations is then repeated. The point of minimum depression of the pencil are obviously a measure of the amount of ammonia (or other constituent to be estimated) in the liquid, and once the instrument has been calibrated the drum can be supplied with paper ruled to give absolute readings. The makers of the instrument, J. v. Geldern and Co., Düsseldorf, supply with it a self-acting compensator for variations in temperature and barometric pressure. This attachment is not shown in the figure. G. C. J.



Constant Flow Aspirator. J. F. Hoffmann. (*Zeitsch. angew. Chem.*, 1912, **25**, 1723-1724.)—A Mariotte aspirator, when operated at a low speed for long periods, often works irregularly on account of the accumulation of gas bubbles in constricted portions of the apparatus. The apparatus here figured overcomes this difficulty. The horizontal limb of the T-tube, which is conveniently constructed from a broken burette, passes through a rubber stopper in the tubulus of the aspirator (not shown in the illustration). Through a rubber collar, a capillary of suitable dimensions is pushed up into the vertical limb of the T-tube, and the advantage of having the latter constructed from a graduated tube lies in the fact that the speed of flow may be regulated by raising or lowering the capillary, and the graduated tube facilitates the regulation. Before setting the apparatus in operation, the bulb which closes the top of the burette is partly or wholly filled with water by suction, and its limb closed by means of a piece of rubber tube and a glass plug. Any bubbles of gas which may escape from the water accumulate in the bulb, and do not interfere with the regular flow of water.

G. C. J.

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REVIEWS.

METHODS OF ORGANIC ANALYSIS. By H. C. SHERMAN. Second edition. New York. 1912. The Macmillan Co. Pp. xvi + 407. Price 10s. 6d. net.

The descriptions of the methods in this book were written primarily for the use of third-year students, and, consequently, the technical analyst will find comparatively little that will assist him. In some respects the work is quite up to date. For instance, the methods of glycerin analysis are those of the International Committees, but in others the methods given are nearly obsolete; and as an instance butter may be cited, the Reichert-Meissl method being described in full, while probably the modification of Polenske is now universally employed.

The best feature of the book is the full and clear descriptions of the methods, which are followed by explanatory notes, and the list of references for the student who wishes to follow up the subject. The work appears admirably designed for the purpose for which it was written.

H. DROOP RICHMOND.

WATER ANALYSIS FOR SANITARY AND TECHNICAL PURPOSES. By HERBERT B. STOCKS, F.I.C. 1912. Charles Griffin and Co. Price 4s. 6d. net.

In his preface the author states that he "has endeavoured to give a concise and accurate description of the methods adopted for the analysis of waters for sanitary and technical purposes, with the hope that the information may be of use to Public Analysts, Medical Officers of Health, and students who are interested in this branch of analytical work."

The volume contains 136 pages, of which twenty-eight are devoted to the organic carbon and nitrogen process of Frankland. Neither the arrangement nor treatment of the subject-matter is original, and although most of the ordinary processes are described, the details and hints given are, in several cases, hardly sufficient to enable those unacquainted with water analysis to obtain the most accurate results. The illustrations are but few, and more than half of them poor in quality. The "apparatus for estimating free and saline ammonia," figured on p. 10, probably compares unfavourably with that to be found in most water-analysis laboratories, and the illustration of the combustion apparatus, given on p. 30, is faulty at one point, and the simple gas-measuring tube indicated would be practically useless for the analysis of the products of combustion of many water residues.

Although the work professes to cover analysis for "technical purposes," it contains no reference to the chemistr softening processes. The examination of sewage and effluents is frequently mentioned, but the methods favoured in the laboratory of the Royal Commission on Sewage Disposal,* are not described, and, although a table of standards for effluents is given, the proposed standards of the Commission are ignored.

From a careful perusal of the volume, it appears that Public Analysts and others must still remain contented with some of the works on water analysis already on their bookshelves. WILLIAM T. BURGESS.

TABLES ANNUELLES INTERNATIONALES DE CONSTANTES ET DONNÉES NUMÉRIQUES. Vol. I. Année, 1910. Gauthier, Villars, Paris; Akademische Verlagsgesellschaft, m. b. H., Leipzig; J. and A. Churchill, London; University of Chicago Press, Chicago. 1912. xxxix + 727. Cloth. 24s. net.

The need of collecting physical data has made itself increasingly felt, and probably the chemist has experienced the want of a handy book of reference even more than the physicist. The latter is concerned, generally speaking, more with the methods by which a property is determined, the limits of accuracy attainable, and, above all, the correlation with other properties; the chemist, on the other hand, usually has in view the connection between the constitution of certain chemical compounds and some particular property. The physicist may have considerable trouble in searching scientific literature with regard to some physical property, but it is light when compared with that of the chemist, who, having some particular group of compounds under review, wishes to compare the values of some particular property. He has to find out for which of the compounds in question the value has been determined; secondly, when discrepant values of the constant are given by different observers, to judge which value is the more reliable. In fact, whilst methods for determining some constant will be found described in a comparatively small number of researches, the values of this constant for different substances may be scattered throughout chemical literature.

Organic chemistry has its "Beilstein" and "Richter," physical chemistry its "Landolt and Börnstein"; the latter does not, however, claim anything like the completeness of the organic works, and to remedy this defect the subject was ventilated at the Seventh Congress of Applied Chemistry, an International Committee nominated, and the table of constants for the year 1910 is now published under the patronage of the Association Internationale des Académies.

Values of constants have been accumulated by a large number of abstractors. From the list it will be noticed that the British Empire and Germany have contributed at least two-thirds of the total. The results have been passed on to a relatively small number (thirty-one) of compilers, all men of established reputation. The British members of this shorter list are Messrs. S. L. Archbutt, H. B. Hartley, A. W. Porter, L. J. Spencer, and N. T. M. Wilsmore. Since the foreign compilers are generally of the same status, it will be seen that the work has been placed in reliable hands.

The principles underlying the general classification are not quite obvious. The titles of the chapters given on p. 21, and the table of contents of pp. 22-39, make it easy to search for any particular property; but why diffusion, degree of association, solubility, and similar matters should be dealt with after optical, electrical, and magnetic properties have been given, and be separated completely from coefficients of compressibility, density and viscosity, with which the work opens, passes the comprehension of the reviewer. Again, details relating to crystallography and mineralogy are sandwiched in between adsorption and organic chemistry, whilst mechanical and other properties of metals and alloys come later in the book. No harm is done, for material is found without much difficulty; but the lack of arrangement is to be regretted in a work on which so much pains has been bestowed.

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* Fourth Report, Vol. IV., Part V., 1904.

The data given are at least as full as one would have expected, and the attempt has been made to record most of the significant figures. Taking a case at random, the densities of solutions of lithium nitrate were determined by Appleby in connection with work on the viscosities of aqueous solutions of this salt, and no less than forty-nine density measurements taken from his work are recorded, in addition to a single value given by Rimbach and Wintgen.

A wise discretion has been exercised in the metallurgical portion; the results of tests (tensile, torsion, etc.) are only recorded for materials of which an analysis has been made. A few matters which had escaped notice in the main part of the work are dealt with in a short supplement.

No index is given; the table of contents renders this scarcely necessary. The care taken in abstracting and compiling is worthy of the highest praise, and the printing and general get-up are excellent. J. T. Hewitt.

NOVEMBER, 1912 Vol. XXXVII., No. 440.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The ordinary meeting of the Society was held on Wednesday evening, November 6, in the Chemical Society's Room, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. Thomas Rigby Greenough, B.A. (Cantab.), A.I.C., "Beechwood," Leigh, Lancs, Assistant Analyst in the City Laboratory, Liverpool; Harold Lowe, M.Sc. (Manchester), A.I.C., 18, Hough Green, Chester, Assistant to Mr. W. F. Lowe, A.R.S.M., F.I.C.; James P. Ogilvie, "Homedale," Hendon Lane, London, N., analytical and consulting chemist; Stanley Elliott, A.I.C., "Penshurst," Windmill Lane, Enfield, assistant to Mr. C. H. Cribb, B.Sc., F.I.C.; and John Algernon Lacy Sutcliffe, A.I.C., City Analyst's Laboratory, Broad Street, Birmingham, assistant to Mr. J. F. Liverseege, F.I.C., were read for the first time.

Messrs. S. W. Collins, B.Sc., F.I.C., and C. B. Lessner, were elected members of the Society.

The following papers were read: "Note on an Apparatus for Testing Water by Measurement of its Electrical Conductivity," by L. Archbutt, F.I.C.; "The Detection and Estimation of Arachis Oil," by Norman Evers, B.Sc.; "The Examination of Chinese and Japanese Wood Oil," by A. Chaston Chapman, F.I.C.; and "The Estimation of Manganese by the Bismuthate Method," by H. F. V. Little, A.R.C.S., B.Sc.

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POISONOUS GASES FROM OILFIELDS.

By HERBERT S. SHREWSBURY, F.I.C.

Recently a negro was killed by breathing the gas at the bottom of a pit under an oil derrick close to the Pitch Lake at La Brea, Trinidad. The pit was some 10 feet deep and surrounded the casing of the tubular oil-well, from which the gas most probably overflowed into the pit. Whilst working in this pit the man became giddy and had to be helped out. Twelve hours afterwards he died.

A sample of gas taken from the top of the well, or oil-tube, was forwarded to this laboratory, and the author found its composition to be as follows:

Sulphuretted hydrogen	0·1
Carbon monoxide	1·1
Saturated hydrocarbons	4·4
Unsaturated hydrocarbons	2·5
Oxygen	8·7
Carbon dioxide	11·9
Hydrogen	18·8
Nitrogen	52·5
	100·0

Assuming all the oxygen and some of the nitrogen to be derived from air, the amount of air present was found to be 43·4 per cent., and the composition of the gas as it issued from the ground was calculated as:

Sulphuretted hydrogen	0·2
Carbon monoxide	1·9
Unsaturated hydrocarbons	4·4
Saturated hydrocarbons	7·8
Carbon dioxide	20·9
Hydrogen	31·4
Nitrogen	33·4
	100·0

A. Wynter Blythe states that 0·05 per cent. of sulphuretted hydrogen in air is the utmost that can be breathed with safety, that 0·08 per cent. is dangerous, and 0·10 to 0·15 fatal to human life. According to Haldane, more than 0·15 per cent. of carbon monoxide is dangerous to human life, and 0·4 per cent. would kill in a moderately short time.

It will be seen that the gas, as it issues from the ground, is extremely poisonous, and that even when mixed with 43 per cent. of air it would kill when breathed for a short time—as, indeed, it did.

The author has visited the Pitch Lake, and the following observations may be of interest. The surface of the lake is covered with little pools and channels of water, through which gas is constantly bubbling. Gas also issues from the pitch itself, which is generally as firm as asphalt, but in places is soft, and in fact liquid. The gas has exactly the same odour as the sample described, the small percentage of sulphuretted hydrogen mixed with the hydrocarbons producing a sweet and tar-like smell. It is an interesting fact, which the author has personally witnessed, that small fishes live in the pools through which the gas bubbles, notwithstanding its poisonous character.

In view of the probability of this evolution of poisonous gas from oilfields being a phenomenon of common occurrence, a widespread publication of the above facts might be the means of averting loss of life.

Government Laboratory,
Trinidad, B.W.I.

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THE DETECTION AND ESTIMATION OF ARACHIS OIL.*

By NORMAN EVERS, B.Sc., A.I.C.

(*Read at the Meeting, November* 6, 1912.)

Part I.—Detection.

J. Bellier (*Ann. Chim. anal.*, 1899, **4**, 4) suggested the following qualitative test for arachis oil in olive oil:

One c.c. of the oil is saponified with 5 c.c. of alcoholic potash (85 grms. potash dissolved in a litre of 70 per cent. alcohol), 1·5 c.c. of acetic acid of such a strength as to exactly neutralise the alcoholic potash is added, and the tube cooled in water below 20° C. for not less than thirty minutes. The liquid is then diluted with 50 c.c. of 70 per cent. (by volume) alcohol containing 1 per cent. (by volume) of hydrochloric acid, and placed in water at 17° to 19° C. for an hour. When the oil contains more than 10 per cent. of arachis oil a precipitate is formed, while pure olive oils give a clear solution.

I have used this test on many olive oils and mixtures of olive oil with arachis oil, and have found it to give quite satisfactory indications with as little as 5 per cent. of arachis oil. As noted by Archbutt (*J. Soc. Chem. Ind.*, 1911, **30**, 5-6; Analyst, 1911, **36**, 116), certain neutralised residuum olive oils, known as "saponified" oils, which contain a high percentage of unsaponifiable matter, give a precipitate with this test.

The test was modified by Mansfeld (*Zeitsch. Untersuch. Nahr. u. Genussm.*, 1905, **17**, 57), Adler (*ibid.*, 1912, **24**, 676), and Franz (*Beiträge z. Nachweis und z. Kenntniss d. Erdnussoles Diss. München*, 1910). A more strongly alcoholic potash solution (80 grms. potash dissolved in 80 c.c. of water and diluted to a litre with 90 per cent. alcohol) is used, and the oil saponified in a flask with a condensing-tube for four minutes. This obviates a difficulty which I have frequently found with Bellier's test—viz., the loss of all the alcohol before the saponification is complete. After adding 1·5 c.c. of dilute acetic acid (1 volume glacial acetic acid to 2 volumes water) and 50 c.c. of 70 per cent. alcohol, the liquid is cooled to 15·5° C., and allowed to remain at this temperature for five minutes. In this way the long standing in cold water is dispensed with. I have found this test, for these reasons, to be preferred to that of Bellier. A distinct turbidity is produced by 5 per cent. of arachis oil, and, except in the case of a sample of "saponified" oil, I have never found a pure olive oil which gave a positive result.

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* This work was carried out under the Analytical Investigation Scheme.

PART II.—ESTIMATION.

Franz and Adler (*loc. cit.*) have shown that the above qualitative test can be made approximately quantitative by determining the temperature at which a turbidity is first produced. The following table is taken from their paper, and shows the various "temperatures of crystallisation" of olive and arachis oils and of mixtures:

Oil.	Temperature of Crystallisation.	Oil.	Temperature of Crystallisation.
	° C.		° C.
Olive oil	11·8–14·3	Arachis oil, 50 per cent.	33·8
Arachis oil, 5 per cent.	15·9–17·0	" 60 "	35·3
" 10 "	19·8	" 70 "	36·6
" 20 "	25·7	" 80 "	38·0
" 30 "	29·2	" 90 "	39·3
" 40 "	31·5	Arachis oils	40·0–40·8

I have been able to verify these temperatures given by Adler with several olive and arachis oils and mixtures, and have found the values obtained remarkably constant, and in all cases within the limits given by him. In the case of the "saponified" olive oil mentioned above, a faint turbidity was observed as high as 45° C., becoming more definite as the liquid was cooled; but no distinct precipitate could be seen until after standing at 15·5° C. for five minutes. This is quite unlike the behaviour of arachis oil, which gives a definite precipitate very shortly after the first opalescence is observed.

Bellier's Process.—This process is carried out as follows:

Five grms. of the oil are weighed out, saponified with 25 c.c. of alcoholic potash (85 grms. potash dissolved in a litre of 70 per cent. alcohol), and the fatty acids liberated with 7·5 c.c. of dilute acetic acid, which exactly neutralises the potash. The liquid is then cooled rapidly and allowed to stand for an hour to precipitate the fatty acids. After filtering, the fatty acids are washed at 15° to 20° C. with 70 per cent. alcohol containing 1 per cent. (by volume) of HCl until the filtrate gives no turbidity with water. The precipitate is then dissolved in 25 to 50 c.c. of boiling alcohol of 92 to 93 per cent. strength, and sufficient water added to reduce the strength to 70 per cent. The fatty acids are allowed to crystallise below 20° C. for an hour, then filtered off, washed with 70 per cent. alcohol until no turbidity is given with water, dissolved in warm ether, and weighed. Using this method, Bellier obtained 4·2 per cent. of fatty acids from two arachis oils, and none or practically none from several olive oils.

Archbutt (Allen's "Commercial Organic Analysis," 4th edition, vol. ii., p. 101) states that he has obtained low results by this method.

The process was tested on various oils in conjunction with Renard's process as modified by Archbutt (*ibid.*, p. 93). The results are given in the following table :

TABLE I.

Oil.	Renard.			Bellier.		
	Arachidic Acid. per Cent.	Melting-point. ° C.	Arachis Oil. per Cent.	Arachidic Acid. per Cent.	Melting-point. ° C.	Arachis Oil. per Cent.
Arachis (A)	4·59	73·5	—	3·56	71	78
Arachis (B)	5·15	72·0	—	3·76	71	83
Olive oil, Nice superfine ...	0	—	—	0	—	—
Olive oil, "Seconds" ...	0	—	—	Trace	—	—
Olive oil, Malaga ...	0	—	—	Trace	—	—
Olive oil, 50 per cent. Arachis (A), 50 per cent.	2·28	73·5	50	1·36	72	30

The factor used in the calculation of the percentage of arachis oil by Bellier's process is 22, as used by Bellier. It is obvious that the results obtained are exceedingly low. This may be due either to the solubility of arachidic and lignoceric acids in 70 per cent. alcohol, or to the incomplete precipitation of the fatty acids on account of their solubility in the strong solution of oleic and other fatty acids.

Solubility of Arachidic and Lignoceric Acids in 70 *per Cent. Alcohol.*—Renard (*Compt. rend.*, **73**, 1330) states that these acids are quite insoluble in 70 per cent. alcohol, but the following determinations show that they possess a quite marked solubility:

The acids employed were obtained from arachis oil by Renard's process. The solubility was determined by dissolving about 0·2 grm. of fatty acid in 100 c.c. of 93 per cent. alcohol, adding sufficient water to reduce the strength to 70 per cent., and keeping at a fixed temperature for several hours. The liquid was then filtered at the same temperature, a measured volume of the filtrate evaporated to dryness, and the residue weighed.

The following are mean values of several results obtained :

TABLE II.

Melting-point.	Solubility, Grms. per 100 c.c.	
	At 13° C.	At 18° C.
71° C.	0·016	0·023
72° C.	0·012	0·017
73° C.	0·009	0·012

The solubility was also determined when the fatty acids were washed on a filter-paper, about 0·2 grm. being used. The results obtained were—

Melting-point, ° C.	Grms. dissolved per 100 c.c.
71	0·008
72	0·006
73	0·005

The presence of 1 per cent. of hydrochloric acid in the alcohol does not affect the solubility.

Saponification, etc.—It is preferable to use the alcoholic potash recommended by Adler, as in the qualitative test, and to saponify in a flask under a reflux condenser.

As a slight excess of acetic acid does not affect the precipitation, 7·5 c.c. of a solution of 1 volume glacial acetic acid in 2 volumes water are added instead of exactly adjusting the strength to that of the potash.

Bellier states that in the absence of 1 per cent. of hydrochloric acid from the 70 per cent. alcohol, the washing is very tedious. The reason does not seem obvious, but this is undoubtedly the case, though the solubility of the arachidic and lignoceric acids themselves is not affected by this amount of hydrochloric acid.

Crystallisation from 90 per Cent. Alcohol.—The final crystallisation was carried out from 90 per cent. instead of 70 per cent. alcohol, where the quantity was sufficient, and the crystals washed, first with 90 per cent., and then with 70 per cent. alcohol as in Renard's process. This procedure gives a purer acid of higher melting-point.

Correction for Solubility in 70 per Cent. Alcohol.—The process was carried out with the above modifications, and corrections added separately for the volume of 70 per cent. alcohol, used for crystallisation and washing, from Table II. above. It was found, however, that when the proportion of arachis oil was less than 50 per cent. the results obtained were too high, because the correction varied with the amount of the precipitate. In order to avoid complication the list of corrections given in Table III. has been drawn up, by means of which a single correction is added for the total volume of 70 per cent. alcohol used, and will be found sufficiently accurate for ordinary purposes.

TABLE III.

Weight of Acids. (Corrected for 90 per Cent. Alcohol.)	Correction per 100 c.c., 70 per Cent. Alcohol.		
	Melting-point, 71° C.	Melting-point, 72° C.	Melting-point, 73° C.
Above 0·10 grm.	0·013 grm.	0·008 grm.	0·006 grm.
0·08–0·10 „	0·011 „	0·007 „	0·006 „
0·05–0·08 „	0·009 „	0·007 „	0·005 „
0·02–0·05 „	0·007 „	0·006 „	0·005 „
Less than 0·02 grm.	0·006 „	0·005 „	0·004 „
Factor for conversion of percentage of fatty acids to arachis oil	17	20	22

TABLE IV.

Oil.	Alcohol used for Crystallisation. Per Cent.	Weight of Crystals.	Correction for 90 per Cent. Alcohol.	Correction for 70 per Cent. Alcohol.	Total.	Per Cent.	Melting-point, °C.	Per Cent. of Arachis Oil by Factor.
		Grm.	Grm.	Grm.	Grm.			
Arachis (A)	90	0·160	0·040	0·027	0·227	4·54	73	100
	70	0·218	—	0·065	0·283	5·66	71	96
Arachis (B)	90	0·163	0·045	0·032	0·240	4·80	72	96
	70	0·233	—	0·068	0·301	6·02	71	102
Arachis (C)	90	0·152	0·054	0·034	0·240	4·80	72	96
Arachis (D)	90	0·194	0·033	0·028	0·255	5·10	72	102
Arachis (A), 50 per cent. ...	90	0·056	0·032	0·022	0·110	2·20	73	48
Olive "Nice," 50 per cent. ...	70	0·090	—	0·055	0·145	2·90	71	49
Arachis (A), 35 per cent. ... Olive "Nice," 65 per cent. ...	90	0·045	0·020	0·029	0·094	1·88	71	32
	90	0·029	0·040	0·020	0·089	1·78	72·5	37
	70	0·059	—	0·040	0·099	1·98	71	34
Arachis (A), 20 per cent. ...	90	0·024	0·012	0·019	0·055	1·10	71	19
Olive "Nice," 80 per cent....	70	0·030	—	0·024	0·054	1·08	71	18
Arachis (C), 20 per cent. ...	90	0·012	0·020	0·015	0·047	0·94	72	19
Olive "Malaga," 80 per cent.	70	0·021	—	0·027	0·048	0·96	71	16
Arachis (A), 10 per cent. ...	90	0·009†	0·008	0·008	0·025	0·50	73	11
Olive "Nice," 90 per cent. ...	70	0·008	—	0·015*	0·023	0·46	70	8
Arachis (B), 10 per cent. ...	90	0	—	—	—	0	—	—
Olive "Nice," 90 per cent. ...	70	0·012	—	0·018	0·030	0·60	71	10
Arachis (C), 10 per cent. ...	90	0	—	—	—	0	—	—
Olive "Malaga," 90 per cent.	70	0·011	—	0·016	0·027	0·54	71	9
Arachis (A), 5 per cent. ... Olive "Nice," 95 per cent. ...	70	0·007	—	0·012*	0·019	0·38	—	6·5
Sesame	90	0	—	—	—	0	—	—
	70	0·012	—	—	—	0·24	64	—
Cotton-seed	90	0	—	—	—	0	—	—
	70	0·006	—	—	—	0·12	50–55	—
Olive "saponified"	90	0·014	—	—	—	0·28	64–67	—
	70	0·021	—	—	—	0·42	64–68	—

* In these cases the correction has been added for melting-point 71° C.

† This result was obtained by recrystallising the fatty acids obtained from 70 per cent. alcohol from 10 c.c. of 90 per cent. alcohol.

The Modified Process.—Weigh out 5 grms. of the oil into a saponification flask, add 25 c.c. of alcoholic potash (80 grms. potash dissolved in 80 c.c. water and diluted to a litre with 90 per cent. alcohol), and saponify for about five minutes under a reflux condenser. To the hot soap solution add 7·5 c.c. of acetic acid (1 volume of glacial acetic acid to 2 volumes of water) and 100 c.c. of 70 per cent. alcohol containing

1 per cent. (by volume) of HCl, and cool to 12° to 14° C. for an hour. Filter and wash with 70 per cent. alcohol containing 1 per cent. HCl at 17° to 19° C., the precipitate being broken up occasionally by means of a platinum wire bent into a loop. The washing is continued until the filtrate gives no turbidity with water, the washings being measured. Dissolve the precipitate, according to its bulk, in 25 to 70 c.c. of hot 90 per cent. alcohol, and cool to a fixed temperature between 15° and 20° C. If crystals appear in any quantity, allow to stand at this temperature for one to three hours, filter, wash with a measured volume of 90 per cent. alcohol (about half the volume used for crystallisation), and finally with 50 c.c. of 70 per cent. alcohol. Wash the crystals with warm ether into a weighed flask, distil off the ether, dry at 100° C., and weigh. If the melting-point is lower than 71° C., recrystallise from 90 per cent. alcohol. Add the correction for the solubility in 90 per cent. alcohol as in Renard's process, from the table given by Archbutt (Allen's "Commercial Organic Analysis," 4th edition, vol. ii., p. 94), and also for the total volume of 70 per cent. alcohol used in precipitating and washing (including the 100 c.c. added in the first instance) from Table III.

If there are no crystals from 90 per cent. alcohol, or if they are only in very small amount, add a sufficient quantity of water to reduce the strength of the alcohol to 70 per cent. (31 c.c. water to 100 c.c. 90 per cent. alcohol). Crystallise at 17° to 19° C. for an hour, filter, wash with 70 per cent. alcohol, and weigh as before, adding the correction for the 70 per cent. alcohol from Table III. If the melting-point is below 71° C., recrystallise from a small quantity of 90 per cent. alcohol, or again from 70 per cent. alcohol.

The results obtained by this method are given in Table IV.

The following oils gave no crystals: Olive oils, including "Nice superfine," "Nice seconds," "Malaga," and eight of unknown origin, almond, poppy, and rape oils.

In conclusion, I wish to express my thanks to Mr. L. Archbutt for his valuable advice and assistance throughout the work.

* * * * *

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Insoluble Carbohydrates of the Apple. E. C. Schneider. (*Amer. J. Physiol.*, 1912, **30**, 258; through *Chem. Zentralbl.*, 1912, II., 937.)—Marc obtained from pounded Ben Davis apples by extraction with water, alcohol, and ether contained 28 per cent. of reducing sugar, 28 per cent. of pentosan, and 11·5 per cent. of galactan; the pectin prepared from this marc by hydrolysis with 0·4 per cent. hydrochloric acid at a temperature of 38°C. contained 57·5 per cent. reducing sugar, 36 per cent. pentosan, and 46 per cent. galactan. Intestinal bacteria destroyed the hemicellulose and pectin; reducing sugars were not found as end-products of this action. Pentosan and galactan were about equally attacked. Marc and pectin were not hydrolysed by animal enzymes, malt diastase, or taka diastase. Experiments on human beings showed that 79 to 84·5 per cent. of the hemicelluloses of the marc were utilised or destroyed during digestion. O. E. M.

Detection of Cane Sugar. S. Rothenfusser. (*Chem. Zeit.*, 1912, **36**, 1100-1101.)—With the exception of cane sugar and raffinose, all sugars are completely destroyed when treated with hydrogen peroxide in alkaline solution (*Cf.* ANALYST, 1910, **35**, 539; 1911, **36**, 588), and the presence of cane sugar in the resulting solution may be ascertained by means of the diphenylamine reagent described previously by the author (ANALYST, 1910, **35**, 208). The use of calcium or barium hydroxide is preferable to that of an alkali hydroxide, as in the former case a precipitate of barium peroxide is formed on the addition of the hydrogen peroxide, and oxygen is liberated quite slowly, so that the sucrose present is not attacked during the digestion. At the end of the operation the excess of hydrogen peroxide in solution is destroyed by the addition of a catalyst, and the solution is tested with the diphenylamine reagent.

W. P. S.

Estimation of Cane Sugar in Foods, etc. A. Jolles. (*Chem. Zeit.*, 1912, **36**, 1100.)—In the method described originally by the author (ANALYST, 1911, **36**, 28) it was recommended that not more than 2 per cent. of sugars other than cane sugar should be present in the solution in which the latter sugar is to be estimated, as with larger quantities the solution becomes brown in colour and the polarimetric examination is affected. He now states that the brown coloration which develops when concentrated sugar solutions are employed in the process may be removed by means of mercuric acetate and phosphotungstic acid. After the sugar solution has been heated with $\frac{N}{5}$ sodium hydroxide solution for twenty-four hours at 37° C., a measured portion of it is neutralised with dilute acetic acid, and 50 per cent. mercuric acetate solution is added as long as a precipitate is formed, the quantity added being noted. The mixture is then filtered, and an aliquot portion of the filtrate is precipitated with a 25 per cent. phosphotungstic acid solution, the necessary volume of which is also noted. The mixture is again filtered, and then examined with the polarimeter. The rotation observed is due solely to cane sugar, and in calculating the quantity of the sugar present, allowance is made for the dilution caused by the introduction of the precipitants. W. P. S.

Analysis of Commercial Casein. W. Hopfner and H. Burmeister. (*Chem. Zeit.*, 1912, **36**, 1053-1054.)—Good commercial casein should exhibit at the most a slight yellow colour, and should be free from rancid or cheese-like odour and from foreign matters. For the estimation of the moisture, 5 grms. of the sample are dried at a temperature of 100° to 105° C. until constant in weight; this requires about two hours' heating, and the weighing should be made at frequent intervals to avoid oxidation of the casein by prolonged heating. The quantity of fat is best estimated by extraction in a Soxhlet apparatus by a mixture of ether and petroleum spirit, the casein being ground and mixed with sand before being placed in the cartridge. A silica basin should be used for the estimation of the ash, and in cases where the ignition proceeds with difficulty the carbonised mass must be extracted with water in the usual way. The acidity of the sample is estimated under the following conditions: Ten grms. of the casein are shaken with 100 c.c. of water, filtered, and 50 c.c. of the filtrate titrated with $\frac{N}{10}$ potassium hydroxide solution, using phenolphthaleïn as indicator. It is useful to determine the solubility of the casein in ammonia. For this purpose 10 grms. of the sample are mixed with 50 c.c. of water and 2 c.c. of 33 per cent. ammonia. After the lapse of a few hours the mixture is warmed to 60° C. Pure casein swells and yields a thick, opalescent liquid, whilst casein which has been stored for a long time gives a turbid liquid. If sand is present it settles to the bottom of the liquid and may be separated and weighed; foreign substances, such as starch, etc., may be detected by means of the microscope. The total nitrogen is estimated by the Kjeldahl method; as regards the calculation of nitrogen into casein, attention is drawn to the different factors, varying from 6·34 to 6·99, which have been proposed. The author finds that the factor 6·61 probably is most correct, as the quantity of casein calculated by its use generally approximates 100 per cent. The maximum and minimum results obtained on the analysis of thirteen samples of commercial casein were: Water, 7·27, 10·50; fat, 0·28, 2·06; ash, 3·53, 4·95; nitrogen, 12·52, 13·55; nitrogen in the fat and ash-free, dry substance, 14·68, 15·65 per cent. W. P. S.

Edible Beans from Hong-Kong. (*Bull. Imp. Inst.*, 1912, **10**, 235-239.)—The following are analyses of three varieties of beans from Hong-Kong, the figures yielded by the corresponding varieties of Indian origin being given in each case:

	Hong-Kong.	Indian Lablab Beans.	Hong-Kong.	Indian Catiang Beans.	Hong-Kong.	Indian "Mung" Beans.
	Dolichos Lablab, Linn.		*Vigna Catiang*, Walp.		*Phaseolus Mungo*, Linn.	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Moisture	12·63	12·1 to 14·6	11·65	12·7	11·88	10·1 to 11·4
Total nitrogen as crude proteins ...	19·51	17·1 to 22·4	22·05	23·1	19·98	22·2 to 23·8
True proteins ...	18·46	—	20·38	—	18·04	—
Other nitrogenous substances ...	1·05	—	1·67	—	1·94	—
Fat	1·24	1·4 to 2·3	1·23	1·1	0·75	2·0 to 2·7
Starch, etc.	57·66	54·2 to 57·4	57·99	55·3	58·96	54·1 to 55·8
Fibre	5·89	5·0 to 6·5	3·83	4·2	4·76	4·2 to 5·8
Ash	3·07	3·4 to 3·6	3·25	3·6	3·67	3·8 to 4·4
"Nutrient ratio" ...	1 : 3·10	1 : 2·5 to 1 : 3·7	1 : 2·75	1 : 2·5	1 : 3·03	1 : 2·5 to 1 : 2·7
"Food units" ...	109·5	105·9 to 113·7	116·2	115·8	110·8	116·3 to 119·3

The "nutrient ratio" is the ratio between the percentage of crude proteins and the sum of the percentages of starch and fat, the latter being first converted into its starch equivalent. The "food-units" is the total obtained by adding the percentage of starch to 2·5 times the sum of the percentages of fat and crude proteins.

A. R. T.

Estimation of Fat in Cocoa by Means of Zeiss' Refractometer. O. Richter. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 312-319.)—The cocoa powder is treated with trisodium phosphate solution and a mixture of ether and alcohol, and the amount of fat present is determined from the refraction of the ethereal solution. The trisodium phosphate solution should contain 65 grms. of the salt per litre, and the alcohol-ether mixture is prepared by mixing equal volumes of these solvents. Preliminary experiments must be made to ascertain how much of the ether-alcohol must be added to 100 c.c. of the phosphate solution so that 5 c.c. of ether shall separate after mixing. The proportions of ether and alcohol in the mixture are so adjusted that about 25 c.c. of the mixture must be added to yield 5 c.c. of ethereal layer. Ten grms. of the cocoa powder are then mixed with this quantity of the ether-alcohol mixture, and 100 c.c. of the phosphate solution are added. The whole is then shaken for some ten minutes and allowed to settle; in the case of chocolate or coarsely powdered cocoa the mixture may be warmed slightly, and then cooled to 17·5° C., at which temperature all measurements of the solutions are made. The clear ethereal layer is next examined in the refractometer, and the percentage quantity of fat (F) present is calculated from the formula:

$$F = \left(\frac{n - n_1}{n - n_2} - 1\right) \frac{100\,xy}{z},$$

where n is the refractive index of cocoa butter (1·4653), n_1 that of the solvent (1·3543), n_2 that of the fat solution observed, x the excess volume of the ether (5 c.c.), y the sp. gr. of the fat, and z the weight of substance in grms. (10 grms.). Tables are also given showing the quantity of fat corresponding with refractive indices ranging from 1·3560 to 1·4200.

W. P. S.

Studies on Flour. R. Fanto. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 269-274.)—With regard to the relation between the gluten-content of a flour and its baking quality, the author points out that rye-flour (which does not yield a coherent cake of gluten although it possesses good baking qualities) does not contain any substances which prevent the coagulation of its gluten or of that of wheat-flour. A mixture of rye- and wheat-flours yields the proportionate quantity of gluten. Rye-flour, therefore, does not appear to contain gluten. The grain obtained by crossing wheat and rye gives a satisfactory cake of gluten. The maximum solubility of the proteins of both wheat- and rye-flours in alcohol is reached when the alcohol has a strength of from 50 to 60 per cent. by volume.

W. P. S.

Composition of the Fatty Oils obtained from the Seeds of *Illicium Verum* Hook and *Illicium Religiosum* Sieb. J. Bulir. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 309-312.)—The fruit of *Illicium verum* Hook (Chinese star-anise) is frequently adulterated with the poisonous fruit of *Illicium religiosum Sieb.* (Japanese star-anise); the fruits are very similar in appearance, but may be distinguished from one another by careful examination. The seeds of both fruits contain a fatty oil; the oils were extracted by the author from specimens of the two seeds by means of ether, then freed from ethereal oils by steam, and dried in an atmosphere of coal-gas. Analyses of the oils yielded the following results:

	Illicium Verum.		*Illicium Religiosum.*	
	Oil.	Fatty Acids.	Oil.	Fatty Acids.
Sp. gr. at 15° C.	0·9264	—	0·9295	—
Saponification value ...	193·8	200·2	193·4	200·7
Iodine value (Hübl) ...	93·1	97·4	90·6	93·6
Hehner value	95·2	—	95·0	—
Reichert-Meissl value ...	1·4	—	1·5	—
Mean molecular weight	—	279·7	—	279·0

In the case of *Illicium verum* the fatty acids consisted of—Oleic acid, 45·0; linolic acid, 23·9; palmitic acid, 23·2; stearic acid, 2·6 per cent. The fatty acid from the oil of *Illicium religiosum* consisted of—Oleic acid, 60·2; linolic acid, 9·8; palmitic acid, 22·5; stearic acid, 2·5 per cent. W. P. S.

Shea-Nuts and Shea-Butter. (*Bull. Imp. Inst.*, 1912, **10**, 281-292.)—The shea-nut is the product of *Butyrospermum Parkii*, found in West Africa and Uganda, etc. The figures yielded on analysis by the "butter" prepared by the natives, and by the fat extracted from dried kernels by petroleum ether, are as follow:

	Sp. Gr., $\frac{99^\circ}{15^\circ}$ C.	Saponification Value.	Iodine Value. Per Cent.	Insoluble Fatty Acids. Per Cent.	Unsaponifiable Matter. Per Cent.	Titer Test. ° C.	Percentage of Fat in Kernels.
SHEA-BUTTER from—							
Lagos	0·862	179·0	58·0	96·5	1·7	52·0	—
Gold Coast, 1	—	184·6	56·0	—	—	51·8	—
" " 2	—	183·0	56·7	—	—	—	—
Bahr-el-Ghazal	—	181·7	54·0	—	—	53·2	—
FAT EXTRACTED from kernels from—							
Southern Nigeria, 1 ...	0·869	181·5	62·0	91·2	6·3	—	48·6
" " 2 ...	0·867	182·8	57·7	94·6	7·0	—	52·4
" " 3 ...	—	181·2	59·4	—	—	—	41·4
" " 4 ...	—	180·2	55·8	—	—	—	46·2
Bahr-el-Ghazal	0·859	184·0	62·9	91·9	4·3	—	47·2

The presence of unsaponifiable matter is accounted for by the presence in the nuts of latex containing a gutta-like body. Any appreciable quantity of this unsaponifiable matter will render the fat less suitable for use in the manufacture of butter substitutes or of soap. The fat appears to contain oleic and stearic acids, and possibly arachidic or lauric acid.

The composition of shea-nut oil-cake is shown below in comparison with other oil-seed cakes:

	Moisture. Per Cent	Ash. Per Cent.	Oil. Per Cent.	Proteins. Per Cent.	Carbo-hydrates. Per Cent.	Crude Fibre. Per Cent.
Shea-nut cake	7·85	5·50	20·30	11·13	48·54	6·68
Cotton-seed cake (decorticated	9·00	7·10	11·38	43·78	23·56	5·18
Cotton-seed cake (undecorticated)	13·75	4·60	6·56	24·62	29·28	21·19
Linseed cake	11·16	5·20	9·50	29·50	35·54	9·10
Soya-bean cake	12·70	5·05	11·07	38·82	26·51	5·85

Shea-nut cake possesses a bitter and unpleasant taste, probably due to the presence of saponin, as is the case with other cakes from sapotaceous oil-seeds. The cake is low in proteins, and, although not actively poisonous, animals do not readily become accustomed to eating it, even in small quantities. A. R. T.

Crude Phytosterol of Cocoanut Oil and the Crude Cholesterol of Butter Fat, and the Detection of Cocoanut Oil in Butter Fat. D. D. Kedrovitsch. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 334-335.)—Six different specimens of cocoanut oil yielded from 0·09 to 0·30 per cent. of crude phytosterol, and the melting-point of the phytosterol acetate varied from 122° to 125° C. Butter fat was found to contain about 0·30 per cent. of crude cholesterol, the acetate of which melted at 113° to 113·5° C. From the examination of the phytosterol-cholesterol acetate obtained from mixtures of cocoanut oil and butter fat, it was found that the presence of 10 per cent. of the former could be detected, the melting-point of the acetate, in this case, lying between 114·8° and 117·5° C. W. P. S.

Solanidine from *Solanum Tuberosum.* **A. Colombano.** (*Gazz. Chim. Ital.*, 1912, **42**, 101-116.)—The solanidine obtained in the hydrolysis of the solanine from the germ of the potato differs in chemical composition and physical properties from the solanidine derived from *Solanum sodomæum.* Specimens of potato solanidine prepared by various methods melted at 214° to 215° C., and had the composition $C_{25}H_{39}NO$ when dried at 105° C. Commercial samples of solanidine and specimens prepared from commercial samples of solanine also had the same composition and melting-point. The solanidine from *S. sodomæum* crystallised in nacreous scales, melting at 197° to 198° C., and had the composition $C_{18}H_{[illegible]}NO$ after drying at 105° C. Another difference between the two compounds is that solanidine from

S. sodomæum does not decolorise an aqueous or chloroform solution of bromine, whereas the potato solanidine forms a fairly stable compound with bromine, melting (with decomposition) at 80° to 90° C. C. A. M.

Polarimetric Estimation of Starch in Sausages. P. Lehmann and E. Schowalter. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 319-327.)—The method proposed depends on the optical rotation of starch in hydrochloric acid solution. Lintner and Ewers have already given values for the rotation of starch in acid solution (*cf.* ANALYST, 1906, **31**, 25; 1907, **32**, 179), but the authors find that commercial wheat-starch has a specific rotation of + 181·1° and potato-starch + 183·15°, under the conditions of their experiments. In the process given below the quantity of substance taken for the estimation and the volumes of the solution are such that each degree of rotation exhibited by the final solution is equivalent to 1 per cent. of starch. The sausage is finely divided, and 27·5 grms. are mixed with 80 c.c. of alcoholic potassium hydroxide solution (100 c.c. of potassium hydroxide dissolved in 100 c.c. of water and diluted with 96 per cent. alcohol to 1 litre) and allowed to stand over-night. The mixture is then boiled under a reflux condenser, and the liquid portion is decanted; the residue is washed once with the alcoholic potassium hydroxide solution, and then with 55 per cent. alcohol, until the washings do not yield a turbidity with hydrochloric acid. Decantation usually suffices in this part of the process, and filtration is seldom necessary. The residue is then dissolved by heating it on the water-bath with 2 c.c. of water and 5 c.c. of 8 per cent. sodium hydroxide solution; the mixture is now neutralised with hydrochloric acid, 25 c.c. of $\frac{N}{1}$ hydrochloric acid are added, and the whole is heated for ten minutes on the water-bath. The mixture is then cooled rapidly, neutralised by the addition of 12·5 c.c. of $\frac{N}{2}$ sodium hydroxide solution, and filtered, the filtrate being collected in a 100 c.c. flask. The precipitate obtained by mixing 3 c.c. of basic lead acetate solution with 6 c.c. of 10 per cent. ammonia is added, the whole is diluted to 100 c.c., filtered, and the filtrate is examined in a 220 mm. tube in the polarimeter. If a 200 mm. tube is employed, 25 grms. of the material should be taken for the estimation. W. P. S.

Lintner's Polarimetric Method for the Determination of Starch. C. E. G. Porst and H. A. Crown. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 13, 213-218.)—The authors have investigated the effect of varying the following conditions: (1) Temperature of digestion with concentrated hydrochloric acid; (2) time of digestion; (3) time of standing after adding the phosphotungstic acid before the reading is taken; (4) volume and concentration of phosphotungstic acid. Results are concordant when the same conditions are observed, and the following method is recommended: Five grms. of starch, or a weight of the finely ground substance equivalent to 5 grms. starch, are mixed with 20 c.c. of water in a mortar cooled down in ice-water. To this is added 40 c.c. of concentrated hydrochloric acid, previously cooled down in a freezing mixture, and the solution is kept at 20° C. for half an hour. The contents of the mortar are transferred to a 200 c.c. flask, 10 c.c. of a 4 per cent. phosphotungstic acid solution added, and made up to volume at 20° C. with acid of 1·125 sp. gr.

The flask is then kept for half an hour in a water-bath at 20° C., filtered, and exactly fifteen minutes after filtering the reading is taken at 20° C. Dry maize starch was thus found to have an $[\alpha]_D$ 199·4. In applying the method to the determination of starch in maize kernels a correction must be applied for the water-soluble matter present. Good agreement was obtained when using the acid method as compared with the diastase and Sachsse methods. In materials such as gluten meal rich in proteins it was found that the readings increased up to a point with the amount of phosphotungstic acid employed to precipitate the protein. The maximum reading was obtained with an amount equivalent to 50 c.c. of the 4 per cent. solution. If more than this were added, the reading fell owing to precipitation of starch. Even with this material, which contained nearly 50 per cent. of protein, the starch value obtained was 44·7 (using the maximum reading), as against 45·2 per cent. by the Sachsse method. The whole operation only takes one hour and a quarter, and if the temperature is kept at 20° C. no inconvenience arises from acid fumes. (*Cf.* ANALYST, 1909, **34**, 332.)

H. F. E. H.

Examination of Storax. C. Ahrens. (*Zeitsch. öffentl. Chem.*, 1912, **18**, 267-272; through *Chem. Zentralbl.*, 1912, II., 873-874.)—A method of examination is based upon the fact that storax is partially soluble in petroleum spirit. From 4 to 5 grms. of the sample are mixed with about 5 grms. of sand and repeatedly triturated with cold petroleum spirit (sp. gr. 0·65; b.-pt., up to 70° C.), the successive extracts being decanted through a filter, until in all about 350 to 400 c.c. of the solution have been obtained. This is distilled in a tared flask and the residue dried in a hot oven until constant in weight. It should be light yellow in colour, strongly refractive, very viscous, and have a pleasant aroma. Pronounced adulteration is indicated by the residue being darker in colour and more solid, and by its odour. This residue is now dissolved in about 30 c.c. of cold neutral alcohol (95 per cent. by vol.) and the solution titrated with $\frac{N}{5}$ alcoholic potassium hydroxide solution, with phenolphthaleïn as indicator, to obtain the acid value; after which an additional 25 c.c. of the alkali solution are added, the flask closed and allowed to stand for twenty-four hours, and the excess of alkali titrated to obtain the saponification value. The moisture is estimated by triturating 4 to 5 grms. of the storax with anhydrous sodium sulphate and ether, and filtering the liquid through a tared paper. The filtrate and washings are made up to 100 c.c., then dried further by means of anhydrous sodium sulphate, and 50 c.c. thereof evaporated in a tared flask. The sodium sulphate is then washed out of the mass on the filter, and the residue dried and weighed. The sum of the two residues deducted from the amount of substance taken gives the amount of water. From the examination of a large number of samples the following limits are provisionally suggested for commercial storax: Petroleum spirit extract, 37·6 to 56·0 per cent. (mean 45·2); acid value of petroleum spirit extract, 33·1 to 62·9 (mean 46·7); and saponification value of petroleum spirit extract, 191·3 to 203·3 (mean 196·1). Products with values outside these limits are to be regarded as inferior or adulterated.

G. A. M.

Tea-Seed Oil and Cake. (*Bull. Imp. Inst.*, 1912, **10**, 234-235.)—The seeds of of *Camellia Sasanqua* (tea-seed) are expressed by the Chinese for their oil, which is used as an illuminant, the residue being employed as a soap-substitute, by virtue of the saponin it contains. Samples from Hong-Kong gave the following results on analysis:

TEA-SEED OIL.

Sp. gr. at 15·5° C.	0·918
Acid value	9·4
Saponification value	193·4
Iodine value per cent.	87·5

TEA-SEED CAKE.

	Per Cent.
Moisture	8·33
Crude proteins	6·49
True proteins	6·13
Other nitrogenous substances	0·36
Fat	1·31
Starch, etc.	43·24
Fibre	37·43
Ash	3·20

The oil sometimes contains saponin, and therefore is unsuitable for edible use. The cake contains 7 to 8 per cent. of saponin, and has an unpleasant and bitter taste, and cannot be used as a feeding-cake. It contains no alkaloids. The amount of saponin is less than is present in quillaia bark, but the cake may be suitable for use in the preparation of vermicides for dressing lawns. A. R. T.

Analyses of Ceylon Tobaccos. (*Bull. Imp. Inst.*, 1912, **10**, 187-205.)—The following are typical examples of analyses of Ceylon-grown tobaccos:

	Samples from Maha-Iluppallama.				Samples from Trincomalee.	
	"Sumatra, No. 1."	"Sumatra, No. 2."	"Java, No. 2."	"Java, No. 3."	December Crop, 1909.	1909-10 Crop.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Moisture	17·5	15·1	12·5	13·2	9·8	15·1
Nicotine	1·8	1·7	1·1	1·8	3·0	1·4
Nitrogen	1·5	3·1	2·0	2·7	3·2	3·3
Ash	16·6	12·6	18·0	15·3	15·6	12·5
Composition of ash:						
Lime	30·7	35·2	30·4	34·1	27·7	33·2
Potash	21·7	19·2	20·3	11·5	16·0	20·1
Soda	0·2	0·4	0·3	1·1	2·4	2·5
Sulphates (expressed as sulphuric acid)	2·4	4·0	4·1	8·4	1·8	4·7
Chlorides (expressed as chlorine) ...	16·1	8·9	10·9	9·1	7·3	6·5
Burning quality ...	Poor.	Poor.	Moderate.	Bad.	Moderate.	Poor.
Nature of ash ...	White; flaky.	White; flaky.	Grey; flaky.	Almost black.	Dark with grey patches.	Black.

A. R. T.

New Colorimetric Method for the Determination of Vanillin in Flavouring Extracts. O. Folin and W. Denis. (*J. Ind. and Eng. Chem.*, 1912, **4**, 670-672.)—The Heis-Prescott method, as modified by Winton (*J. Amer. Chem. Soc.*, 1899, **21**, 256; 1902, **24**, 1,128; 1905, **27**, 719), though accurate, is laborious. By the use of the phosphotungstic-phosphomolybdic reagent (*J. Biol. Chem.*, 1912, **12**, 239) an accurate colorimetric method can be employed, which gives theoretical results with artificial extracts containing known amounts of the substance, and with authentic extracts gives values identical with those obtained by the official method. The solutions required are (1) an aqueous solution of pure vanillin, 10 c.c. of which contains 1 mgrm. of vanillin; (2) phosphotungstic-phosphomolybdic acid prepared as follows: To 100 grms. pure sodium tungstate and 20 grms. phosphomolybdic acid (free from nitrates and ammonium salts) are added 100 grms. of syrupy phosphoric acid (containing 5 per cent. H_3PO_4) and 700 c.c. water: the mixture is boiled over a free flame for one and a half to two hours, cooled, filtered if necessary, and made up to 1 litre. An equivalent amount of pure molybdic acid may be substituted for the phosphomolybdic acid. (3) Saturated sodium carbonate solution. (4) A solution containing 5 per cent. basic and 5 per cent. neutral lead acetate. Vanillin and other mono-, di-, and tri-hydric phenol compounds, when treated with the reagent (2), give on the addition of excess of sodium carbonate solution the deep blue colour which is the basis of the new method. The method cannot be applied to the untreated extract, since tannin also reacts with the reagent, and the following procedure is required: Five c.c. of the extract to be examined are diluted with about 75 c.c. of water in a 100 c.c. flask, 4 c.c. of the lead acetate solution added, and the mixture made up to volume, filtered, and 5 c.c. of the filtrate placed in one 50 c.c. flask, and in another 5 c.c. of the standard vanillin solution. Five c.c. of the phosphotungstic phosphomolybdic reagent are added to each flask, the solution shaken and allowed to stand five minutes, and then each flask is made up to volume with sodium carbonate solution. The solutions are then well mixed and allowed to stand ten minutes, by which time the precipitation of sodium phosphate is complete. The contents of the flasks are filtered rapidly, and the colour of the filtrates, which must be quite bright, are compared in a Dubosc colorimeter. The standard solution is best placed at 20 mm. Thus, if 5 c.c. of the diluted and filtered solution gave a reading of 18 mm., the result would be $0{\cdot}5 \times \frac{20}{18} = 0{\cdot}555$ mgrm., and $55{\cdot}5 \div 250 = 0{\cdot}22$. Hence 0·22 per cent. of vanillin. An advantage of the method is the small amount of solution required, 2·5 c.c. of extract being enough for from two to four duplicate determinations. The official method may be readily checked by using the ether extract after drying and weighing and dissolving it in ethyl alcohol, the estimation then proceeding as already described. Night readings can be taken, the best illumination being an ordinary incandescent electric light, covered with a sheet of white paper. A new portion of the standard solution should be made for each batch of twelve samples examined, as the colour darkens for an hour after the addition of the alkali. A large number of results are recorded determined by the new and the official methods, which show agreements in all cases within 0·01 per cent. Although vanillin, even in dilute solution, may be precipitated with basic lead acetate, in the high dilution above recommended no precipitation occurs. Coumarin, extract of tonka bean, and acetanilide give no colour with the phosphotungstic-phosphomolybdic reagent, and the presence of sugar, caramel, or glycerol does not interfere with or alter the colour formation.

H. F. E. H.

Estimation of Extract in Wort and Beer. W. Fresenius and L. Grünhut. (*Zeitsch. anal. Chem.*, 1912, **51**, 643-658.)—A comparison is instituted between the tables of Balling, Windisch, Schultze, Ostermann, Elion, and Riiber. For the determination of extract in beer, the table of Windisch is shown to give the least inaccurate values. Windisch's table ("Tafel zur Ermittlung des Zuckergehaltes wässriger Zuckerlösungen," 1896) is a cane-sugar table, and, applied to solutions of maltose and dextrin, leads to results much above the truth, as may be calculated from the results of Brown, Morris, and Millar, or those of Ost or Meissl. But in the case of beer extracts the presence of the small amounts of glycerol and non-volatile acid normally present goes far towards compensating the above error, for these substances are under-estimated by 35 to 40 per cent. if the solution factor for cane-sugar be applied to their solution. The nitrogenous constituents of the extract also tend to reduce the *plus* errors attaching to the use of Windisch's table, whilst the ash constituents tend to increase it. The algebraic sum of all the errors is always positive, and appears to lie between 0·4 and 1·2 per cent.

The extract of unfermented malt-worts is over-estimated by as much as 2 per cent. if calculated by means of Windisch's table, since the influence of the maltose and dextrin in unfermented wort quite outweighs the influence of the minor constituents. Of existing tables, that of Riiber ("Videnskabsselskabets Skriften I., Mathematisk-naturv. Klasse," 1897, No. 5) gives the smallest errors when applied to unfermented malt worts. The error is a *minus* one, and its magnitude very close to 1 per cent. Riiber's table being somewhat difficult of access, and occupying several pages in its original form, the present authors have condensed it as follows. The table gives the percentage of extract corresponding to every degree of gravity from 1,000 to 1,085, and the nine columns on the right show the addition to be made for each tenth of a degree.

Extract Table according to C. N. Riiber. (Percentage by Weight.)

d 15°/15°	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
1·00 ..	0·00	0·25	0·50	0·75	1·00	1·25	1·49	1·74	1·99	2·24	02	05	07	10	12	15	17	20	22
1·01 ..	2·48	2·73	2·97	3·22	3·46	3·70	3·95	4·19	4·43	4·68	02	05	07	10	12	15	17	20	22
1·02 ..	4·92	5·16	5·40	5·64	5·88	6·12	6·36	6·60	6·84	7·07	02	05	07	10	12	14	17	19	22
1·03 ..	7·31	7·55	7·78	8·02	8·26	8·49	8·73	8·96	9·20	9·43	02	05	07	09	12	14	17	19	21
1·04 ..	9·67	9·90	10·13	10·36	10·60	10·83	11·06	11·29	11·52	11·75	02	05	07	09	12	14	16	18	21
1·05 ..	11·98	12·21	12·44	12·67	12·89	13·12	13·35	13·58	13·80	14·03	02	05	07	09	11	14	16	18	21
1·06 ..	14·26	14·48	14·71	14·93	15·16	15·38	15·61	15·83	16·06	16·28	02	04	07	09	11	13	16	18	20
1·07 ..	16·50	16·72	16·95	17·17	17·39	17·61	17·83	18·05	18·27	18·49	02	04	07	09	11	13	15	18	20
1·08 ..	18·71	18·93	19·15	19·37	19·59	19·80	—	—	—	—	02	04	07	09	11	13	15	17	20

G. C. J.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Structural Elements of Cellular Membranes. J. König. (*Chem. Zeit.*, 1912, **36**, 1101.)—In addition to true cellulose, the cellular tissue contains—(A) Fat, wax, resin, tannin, aromatic aldehydes, glucosides, etc.; (B) hemicelluloses; (C) lignins; (D) cutin and suberin. The substances under Group A may be removed by treatment with dilute alcohol or with alcohol, benzene, and ether; the Group B by hydrolysis with dilute acids; the Group C by feeble oxidising agents in alkaline solution; and the Group D by treating the residue from the preceding groups with dilute ammoniacal copper solution or zinc chloride and hydrochloric acid, by which the true cellulose is not attacked. The substance obtained by treating a vegetable substance with dilute acids and alkalis, or with glycerol-sulphuric acid, is not cellulose; it contains large quantities of lignin, together with hemicellulose (pentosan), and is more properly termed "crude fibre" than cellulose. The "cellulose" prepared from wood for paper-making contains considerable quantities of pentosan and lignin. By treating rasped wood with dilute mineral acid, followed by oxidation with the usual agents, a comparatively pure cellulose may be obtained, and also by-products which may be used as fodder.

When beech or pine wood, or hemp fibre, has been treated with benzene, alcohol, and water, and then subjected to hydrolysis with 72 per cent. sulphuric acid, subsequent treatment with water yields a clear solution and a brownish-black sediment; the latter has the structure of the cell-membrane, and may be oxidised by hydrogen peroxide in ammoniacal solution. It consists of lignin, and in this way not only the cellulose, but the lignin, may be estimated in cellular tissues. It is not considered probable that the lignin is combined with cellulose in vegetable tissues, as, if this were so, the two substances would not preserve their original structure when separated. It would appear to be likely that the lignin and the cellulose were mixed in the tissue in much the same way that calcium phosphate and proteins exist in bones.

W. P. S.

Delicate Method for Determining Minute Quantities of Chlorophyll. C. A. Jacobson. (*J. Amer. Chem. Soc.*, 1912, **34**, 1266-1269.)—The author has extended the work of Jacobson and Marchlewski (ANALYST, 1912, 460) in the separation of neo- and allo-chlorophyll, and, modifying the photographic process there described, uses it for the estimation of traces of chlorophyll. The intensity of the chlorophyllan absorption bands in the ultra-violet of the solution to be examined is compared with that of corresponding bands produced by solutions of known concentration. Chloroform, as before, is used as the solvent, and 4 or 5 c.c. of solution was found to be sufficient for the purpose, containing as little as 0·02 mgrm. of chlorophyll. A leaf whose green weight is 0·2 grm. suffices for determining the quantity and character (*i.e.*, ratio of neo- to allo-chlorophyll) of the chlorophyll it contains. Eight photographic plates are given, illustrating the variations in intensity of the spectra obtainable. Leaves are macerated in a mortar with cold 92 per cent. alcohol, filtered, and the residue again ground until the extract is colourless. Two c.c. of 10 per cent. alcohol oxalic acid solution is added to the extract, and the whole allowed to stand two or three hours. Calcium carbonate is then added in slight excess, and the mixture evaporated to dryness on the water-bath. The residue is then extracted with chloroform till colourless, and the chloroform solution is diluted appropriately for photographing the spectrum. H. F. E. H.

Nature of Peroxydase. A. Hesse and W. D. Kooper. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 301-309.)—The results obtained during an investigation as to the nature of the substance which yields the "peroxydase" when milk is treated with Rothenfusser's, Storch's, and Arnold's reagents, in the presence of hydrogen peroxide, lead the authors to the conclusion that inorganic substances also play a part in the reaction, and that these substances are not the alkaline constituents of the milk (*Cf.* ANALYST, 1912, 141). They are of the opinion that the colour reaction is due to the catalytic action of iron compounds such as ferrous lactate. This is borne out by the extreme sensitiveness of the reagents towards ferrous lactate, even in very dilute solutions of the latter, and by the fact that the iron solution loses its property of giving a coloration with the reagents when it has been boiled, either alone or in the presence of milk, or even when it has received the addition of boiled milk. Further, substances such as lactic acid, sodium thiosulphate, ethyl alcohol, methyl alcohol, and amyl alcohol, which prevent the so-called peroxydase reaction, also inhibit the action of ferrous lactate on the reagents, whilst mercuric chloride and chloroform (two substances which destroy enzymes) do not interfere with the reaction, either in the case of milk or of ferrous lactate. W. P. S.

Proteolytic Enzyme of Malt. R. Wahl. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 14, 216-229.)—The proteolytic activity of malt extracts is much increased by the presence of bacterial acidity due to lactic acid, and the author concludes that the peptic enzyme contained in the malt is combined with some base of an albuminous nature, and becomes active through liberation by bacterial acidity. The optimum temperature for the splitting of albumin in presence of lactic acid is about 30° C., and the reaction is rapidly brought to a standstill at about 50° C. It has hitherto been stated that the optimum temperature for peptic activity was from 40° to 50° C. The substitution of mineral or commercial acid for lactic acid of bacterial origin does not result in any increased activity, and this latter acid, when added to a brewer's mash, increases the total yield obtainable from the malt, owing to an increase in peptic activity. Commercial malts differ greatly as regards their peptic activity, high-dried malts being less active than low-dried. The lactic acid employed for the acidulation of the water used in the malt extractions was prepared by inoculating a mixture of 1 pint crushed malt in 4 pints of water with a culture of lactic acid, and raising to a temperature of 50° or 60° C. for about twenty-four to forty-eight hours, by which time 1 or 2 per cent. of lactic acid is formed. The grains are then separated and the liquid used in suitable dilution for the extraction of the malts under examination. The peptic strength of the extracts was determined by auto-digestion and by a modified "gelatine liquefying strength" method adopted from that of Schidrowitz (*J. Inst. Brewing*, 1904, **10**, 166). The peptic strength of malt extracts was always compared with that of a standard solution of pepsin of known strength.

The gelatine is prepared as follows: 32 grms. of gelatine are dissolved in 318 c.c. of distilled water by gently warming. One-third of a grm. of finely-ground egg albumin dissolved in 50 c.c. of distilled water is then added, and the gelatine solution cooled to 52° C. The temperature is then raised to 85° C. in five minutes, then kept at 100° C. for ten minutes, and filtered into a beaker containing 0·5 grm. of thymol. Tubes are then filled with 6 c.c. of the gelatine solution, and closed with corks to prevent evaporation. When required for use the tube is gently warmed in a water-bath until the contents are liquefied. Five c.c. of the extract to be examined are added, a blank tube being run alongside with 5 c.c. of water. The tubes are incubated for four hours at 37·5° C., and afterwards placed in ice-water at 2° C., the time required for solidification of the gelatine being noted. The time for the blank tube to solidify is subtracted from that of the other tube, as the lactic acid has a slight liquefying action on gelatine. The pepsin standard is prepared by mixing 0·15 grm. of pepsin of 10,000 strength with 10 grms. of powdered sugar, and dissolving the whole in 100 c.c. of distilled water. Five c.c. of this solution are added to a gelatine tube, and, under the conditions already described, should require seven minutes for solidification.

The auto-digestion method consists in measuring the amount of coagulable albumin remaining in the malt extracts after digestion for a suitable time and temperature, and is carried out as follows: The clear filtered extract, after the above-mentioned digestion, is heated to 75° C., and kept at this temperature for thirty minutes, when it is cooled to 25° C. Ten c.c. of the solution are then centrifuged for four minutes at a speed of about 2,500 per minute. The coagulated albumin is then read in $\frac{1}{10}$ths of c.c. A number of values are recorded with varying amounts of lactic acid acidity, and also with mineral acids, none of the latter showing a solidification time beyond five minutes, as against the fifteen to twenty minutes with 1·5 to 2 per cent. lactic acid. Malts containing moisture from 2 to 3·9 per cent. gave gelatine values of sixteen to twenty-one minutes, while from 4 to 6·4 per cent. of water lowered this to about ten minutes. High extract malts also gave a higher gelatine test indicative of greater peptic strength. H. F. E. H.

Sensitive Test for the Detection of Albumin in Urine. A. Jolles. (*Chem. Zeit.*, 1912, **36**, 1108.)—The reagent employed is prepared by dissolving 10 grms. of mercuric chloride, 20 grms. of citric acid, and 20 grms. of sodium chloride in 500 c.c. of water. Five c.c. of the filtered urine are placed in each of three test-tubes; the first tube then receives the addition of 1 c.c. of 30 per cent. acetic acid, and 5 c.c. of the reagent, the second tube 1 c.c. of the acetic acid. Water is then added to the second and third tubes until they are filled to the same height as the first tube, and their contents mixed by shaking. On placing tube No. 3 between Nos. 1 and 2, a difference in the turbidity of the contents of the two latter is readily detected; in the presence of albumin and pus the contents of tube No. 1 will be more turbid than those of tube No. 2. The presence of mucin and nucleo-albumin may be detected by comparison with tube No. 3; an increase in the turbidity of the contents of tube No. 2 when diluted with water indicates the presence of nucleo-albumin. The test will detect 1 part of albumin in 120,000 parts of urine. W. P. S.

Detection of Blood in Urine and other Physiological Fluids. F. Michel. (*Chem. Zeit.*, 1912, **36**, 994-995.)—The guaiacum test for blood is rendered more sensitive if a small quantity of pyridine is added to the urine or other fluid under examination. Five c.c. of urine should be mixed with about 1 c.c. of pyridine before the ozonized turpentine or hydrogen peroxide is added. The extraction of the urine with pyridine, according to the method described by von Furth for the detection of blood on fabrics (ANALYST, 1911, **36**, 549) is in many cases unsatisfactory, as substances are extracted from the urine which interfere with the subsequent test for blood. The following method of carrying out the test is stated to be more trustworthy, and to be capable of detecting the presence of 1 part of blood in 200,000 parts of urine: Twenty-five c.c. of the urine (filtered, if necessary) are treated with 5 c.c. of a 15 per cent. calcium chloride solution, and heated to boiling. To the hot solution are added 5 c.c. of a 10 per cent. sodium phosphate solution, the precipitate (consisting of calcium phosphate together with coagulated blood) is collected on a filter, washed with a small quantity of physiological salt solution, and a portion of it is transferred to a strong stoppered test-tube. Three c.c. of water, 3 c.c. of pyridine, and a few drops of potassium hydroxide solution are added, and the mixture is boiled. It is then shaken, while hot, with an equal volume of 50 per cent. potassium hydroxide solution, the pyridine layer is separated, again shaken with 50 per cent. solution, and separated. This pyridine solution is now treated with an equal volume of glacial acetic acid, 0·5 c.c. of a leuco-malachite green solution (*Cf.* ANALYST, 1911, **36**, 284), and 2 drops of 1 per cent. hydrogen peroxide. An intense green coloration develops if blood is present. Another portion of the calcium phosphate precipitate may be dissolved in a few drops of glacial acetic acid, treated with a small quantity of pyridine, and then tested with the leuco-malachite green solution in the presence of hydrogen peroxide. In both tests the coloration must develop within two minutes, and be of a deep tint; a faint green coloration, which does not indicate the presence of blood, is given by the calcium phosphate precipitate obtained from most normal urines.

W. P. S.

Estimation of Cane-Sugar in Urine in the Presence of Other Sugars. A. Jolles. (*Biochem. Zeitsch.*, 1912, **43**, 56-64.)—The method described previously by the author (*Cf.* ANALYST, 1911, **36**, 28) may be applied to the estimation of cane-sugar in urine under the following conditions: Urine containing less than 5 per cent. of dextrose is diluted with an equal volume of water, but when the dextrose exceeds 5 per cent. it is necessary to dilute 1 volume of urine with 2 volumes of water. Of the diluted urine, 100 c.c. are treated with 2·5 c.c. of a 16 per cent. sodium hydroxide solution, and then maintained at a temperature of 37° C. for twenty-four hours. After cooling, the mixture is rendered slightly acid with acetic acid, and 50 c.c. are mixed with 5 c.c. of 10 per cent. lead acetate solution, filtered, and the filtrate is examined with the polarimeter. A slightly negative reading (about – 3° Ventzke) indicates that cane-sugar is absent, whilst a positive reading shows that this sugar is present in the urine, and its quantity may be calculated from the reading observed, allowance being made for the extent to which the urine has been diluted. As little as 0·2 per cent. of cane-sugar may be detected in urine by means of the method. Trustworthy results cannot, however, be obtained in the case of urines containing a large quantity of ammonium salts, or of β-hydroxybutyric acid or other optically active substance. W. P. S.

Detection of Lævulose in the Presence of Dextrose. A. Jolles. (*Chem. Zeit.*, 1912, **36**, 1108.)—The author's modification of the Ihl-Peckmann reaction will detect the presence of 0·1 per cent. of lævulose in urine which also contains 5 per cent. of dextrose. The test is carried out as follows: 1 c.c. of diluted urine (1 : 10) is treated with 10 drops of a 20 per cent. alcoholic solution of diphenylamine and 1 c.c. of concentrated hydrochloric acid, and the mixture is boiled for fifty seconds. A comparison test should be made at the same time with normal urine, containing a similar quantity of dextrose. The detection of pathological constituents in urine is rendered difficult owing to the increasingly large number of substances which are administered as medicines. For instance, a sample of urine was found to be lævorotatory, yielded positive reaction with phenylhydrazine and Seliwanoff's test, but failed to give the Ihl-Pechmann reaction, and a fermentation test gave a negative result. This urine was from a patient who, for three weeks, had been taking validol (a compound of menthol with valerianic acid). W. P. S.

Detection of Glycuronic Acid in Diabetic Urine. A. Jolles. (*Chem. Zeit.* 1912, **36**, 1108.)—Since dextrose gives a reaction with naphthoresorcinol, it is not possible to detect glycuronic acid directly in diabetic urines by means of this reagent. The test may, however, be carried out as follows: From 200 c.c. to 400 c.c. of the urine are treated with lead acetate until a precipitate no longer forms; after settling, the clear solution is passed through a filter and the precipitate is washed four successive times by decantation with water, using 400 c.c. each time. The filtrate (excluding the washings) is now precipitated with basic lead acetate, and this precipitate is washed in a similar way. The two precipitates are united, mixed with a small quantity of water, heated to 70° C., the lead is removed as sulphide, and the solution is evaporated to 20 c.c.; 5 c.c. of this solution are then tested with naphthoresorcinol. W. P. S.

Method for the Estimation of Hippuric Acid in Urine. T. Hryntschak. (*Biochem. Zeitsch.*, 1912, **43**, 315-322.)—The method is based on the oxidation of hippuric acid by permanganate, the resulting benzoic acid being separated subsequently by extraction with ether. One hundred c.c. of the urine are boiled with 10 grms. of sodium hydroxide in a flask (about 1 litre capacity) under a reflux condenser for one hour. The contents of the flask are then cooled, the condenser is removed, and potassium permanganate is added, in small quantities at a time, until the liquid has a distinct permanganate tint after a further seven minutes' boiling; in the case of human urine, 10 grms. of permanganate is usually a sufficient quantity. The mixture is again cooled, a few pieces of ice are added, together with about 15 grms. of sodium bisulphite. The condenser is attached to the flask, the latter is placed in a vessel of cold water, and 50 per cent. sulphuric acid is added through the condenser until all the manganese dioxide has dissolved. The flask may now be placed aside for a few hours and the contents then extracted five successive times with ether, the condenser and the flask being also rinsed with the solvent. The united ethereal extracts are dried by the addition of fused sodium sulphate and transferred to a round glass basin, the last portion of the ethereal solution being decanted through a small filter in order to remove fragments of sodium sulphate. After the ether has been evaporated the residue is dissolved in a small quantity of chloroform (previously washed with water and dried over anhydrous copper sulphate), the solution is filtered into a weighed flask, and the chloroform is evaporated by placing the flask in a vessel through which a current of dry air is passed. The weight of the benzoic acid found is multiplied by 1·468 to give the amount of hippuric acid present. Results of experiments are recorded which show that, when hippuric acid is added to urine, from 96·1 to 99·9 per cent. of the amount introduced may be recovered by means of the above-described method, provided that an allowance is made for the quantity of benzoic acid yielded naturally by different urines when subjected to the process. In the case of human urine the benzoic acid found amounts to 0·056 grm. per 100 c.c.; with dog's urine it is 0·009 grm., and for horse's urine 1·007 grms. The presence in urine of such substances as hydroxyphenylacetic acid, hydroxyphenylpropionic acid, phenylaceturic acid, and of benzoic acid contained in a glycocoll complex, will influence the results, but, as a rule, these substances occur in such small quantity that their effect may be neglected.

W. P. S.

ORGANIC ANALYSIS.

Elementary Analysis. A. Kurtenacker. (*Zeitsch. anal. Chem.*, 1912, **51**, 639-640.)—The author recommended (ANALYST, 1911, **36**, 516) the use of reduced nickel in the forward part of a combustion-tube to reduce oxides of nitrogen. Doht (*Zeitsch. anal. Chem.*, 1911, **50**, 755) criticised that paper on the ground that its author rejects the use of metallic silver for the same purpose, without adducing any evidence of its failure to reduce oxides of nitrogen and without reference to the work of Epstein and Doht (*Zeitsch. anal. Chem.*, 1907, **46**, 771), who recommended it. The author now points out that in his original paper he gave references to the work of others who had shown the insufficiency of silver, brings forward experimental evidence of his own to the same effect, and shows that the work of Epstein and Doht is inconclusive, since many of the test substances used by them—*e.g.*, *p*-nitraniline and *m*-dinitrobenzene—give accurate results when submitted to combustion in an open tube without the use of any device for the reduction of oxides of nitrogen.

G. C. J.

Estimation of the Acetyl Value. W. Normann. (*Chem. Rev. Fett- u-Harz-Ind.*, 1912, **19**, 205-206.)—The following rapid method obviates errors caused by esterification of acetylated fatty acids when dissolved in hot alcohol: About 2 grms. of the filtered fat are weighed into a saponification flask and boiled with 4 to 6 c.c. of acetic anhydride for thirty minutes to an hour beneath a reflux condenser. The flask is then immersed in a hot-water bath, while a current of carbon dioxide hydrogen (or air in the case of non-oxidisable fats) is blown through it, until the whole of the residual acetic anhydride has evaporated. The contents are next diluted with a little ether, about 5 c.c. of water added, any remaining traces of acetic anhydride and the free fatty acids neutralised with a few drops of aqueous sodium hydroxide solution, and the fat saponified with 50 c.c. of $\frac{N}{2}$ alcoholic potassium hydroxide solution, the excess of which is titrated with $\frac{N}{2}$ hydrochloric acid. The amount of alkali expressed in the usual way gives the value for the saponification of the glycerol ester and of the acetic ester. By deducting from this value the saponification value of the original fat, if neutral, or the ester value if free fatty acids were present, a value is obtained showing the amount of alkali corresponding to the free hydroxyl groups in the original fat. For this the term *hydroxyl value* is suggested in contradistinction to the acetyl value, which only relates to the acetylated fatty acids.

C. A. M.

Hübener Bromide Method for the Estimation of Caoutchouc. R. Becker. (*Gummi-Zeit.*, 1912, **26**, 1503; through *Chem. Zentralbl.*, 1912, II., 966.)—The Hübener bromide is not entirely soluble in chloroform (*cf.* Hinrichsen and Kindscher, ANALYST, 1911, **31**, 224), but is dissolved completely by a mixture of chloroform and carbon disulphide. Caoutchouc brominated by the method of Budde did not show this peculiarity. The proportion insoluble in chloroform increases with the amount of bromine in excess during the bromination. The substance insoluble in chloroform is of a sticky consistency, and may easily be taken for unconverted caoutchouc. The author considers that the error thus introduced into the Hübener method can be avoided by bringing the rubber into less intimate contact with the bromine; this is effected by placing the bromine in a special stoppered porcelain vessel pierced with fine holes at 5 to 10 mm. from the bottom. This is removed from the beaker when the powdered bromide is returned to it. The bromine may also be the cause of the oxidation of vulcanisation sulphur. The presence of sulphur trioxide in the bromine, mentioned by Hübener, is confirmed.

O. E. M.

Hübener Bromide Method for the Estimation of Caoutchouc. G. Hübener. (*Gummi-Zeit.*, 1912, **26**, 1711; through *Chem. Zentralbl.*, 1912, II., 967.)—The sticky substance insoluble in chloroform, observed by Becker (see preceding abstract), may be due, not to over-bromination, but to the presence of compounds of bromine or oxygen with resins, or of the resins themselves, as Becker does not state that these had been removed from the rubber. Pure tetrabromide is not always obtained by the method of Budde (ANALYST, 1909, **34**, 407). Korneck (*ibid.*, 1911, **36**, 158) found that it often contains enclosures of unaltered rubber, and, as Spence and Galetly have shown (*ibid.*, 1911, **36**, 361), the whole of the protein is present. On the other hand, Utz (*ibid.*, 1912, 361) obtained a bromide containing 70·18 per cent. of bromine—a nearly theoretical result. Regenerated rubber, containing more constituents soluble in acetone, yields more substance insoluble in chloroform than does raw rubber, but this comes not wholly from resin and oil, but also from the caoutchouc-sulphur compound which at present cannot be broken up in the process of regeneration. The author's modified method is as follows: The rubber is rasped fine, and 2·5 grms.

are wrapped in filter-paper and extracted in a Soxhlet apparatus with acetone for from four to six hours. If resin insoluble in acetone is present, as in hard rubber mixtures, an extraction with dichlorhydrin follows. The residue is dried at 60° C., and 0·2 grm., or more if necessary, digested with 100 c.c. of water and 5 to 10 c.c. of bromine at a moderate temperature on the sand-bath, until the rubber is completely opened out and most of the bromine driven off. The rest is removed by boiling. The precipitate is washed on a filter with hot water, dried at 50° to 60° C., and fused at a red heat in a platinum crucible with sodium carbonate free from halogen. The melt is dissolved by warming in aqueous nitric acid; overheating must be avoided. The bromine is then titrated (*Chem. Zeit.*, 1909, **33**, 648). An analysis in which extraction with acetone was omitted gave a result exceeding the theoretical by 3·5 per cent.; this is regarded as satisfactory, as the acetone extract was especially high. The author's method is suitable only for vulcanised rubber. O. E. M.

Estimation of Cellulose in Woods and Textile Fibres. C. F. Cross and E. J. Bevan. (*Zeitsch. f. Farbenind.*, 1912, **11**, 197-198; through *Chem. Zentralbl.*, 1912, II., 1066.)—In a paper by König and Hühn (*Zeitsch. f. Farbenind.*, 1911, **10**, 297), it was contended that Cross and Bevan's chlorination process is one of oxidation by which considerable residues of pentosans and other bodies differing from true cellulose are left in the product, whilst the cellulose itself is appreciably modified by oxidation. König and Hühn recommended in preference a process consisting, first, of a hydrolysis with sulphuric acid dissolved in glycerol, whereby pentosans and hemihexosans are hydrolysed and dissolved, and, secondly, of an oxidising treatment (hydrogen peroxide and ammonia) whereby lignin is removed. The present authors condemn such a method on the ground that the products obtained are residues of hydrolysis and degradation, and bear no definitely ascertained quantitative relationship to the original material. The main characteristic of a "normal" cellulose is its quantitative persistence through a cycle of chemical changes—*e.g.*, (*a*) Nitration-denitration, or (*b*) conversion into xanthogenate-reversion to cellulose, with all its colloidal structural properties unchanged. The chlorination of lignin complexes is a specific reaction, and secondary oxidation changes in the cellulose residue may, under proper conditions, be almost entirely suppressed. The authors do not accept the view that the production of furfural from the celluloses isolated by their method indicates the presence of pentosan non-celluloses as residual "impurities." This is supported by the fact that the celluloses isolated from the Gramineæ, by drastic and purely hydrolytic processes (caustic soda), yield 12 to 14 per cent. of furfural, which it is impossible to regard as being derived from adventitious pentosans. Thus, König and Hühn's contention that cellulose, which contains furfural-yielding groups, has not been sufficiently purified by hydrolysis, is untenable. J. F. B.

Estimation of Cellulose in Woods and Textile Fibres. J. König and F. Hühn. (*Zeitsch. f. Farbenind.*, 1912, **11**, 209-211; through *Chem. Zentralbl.*, 1912, II., 1066-1067.)—The authors reply to the specific points raised by Cross and Bevan (see preceding abstract). They state that Cross and Bevan have not proved that chlorination is unaccompanied by oxidation. They rely on the work of Tollens in support of their contention that furfural-yielding groups are synonymous with pentosans, which they contend must be removed by still more drastic hydrolysis before the cellulose can be considered pure. The maximum specific rotation of solutions of cellulose in acid zinc chloride was not put forward as a test for pure cellulose, but the fact was merely recorded that the solutions which showed the maximum rotations were derived from celluloses which, by other characters, were proved to be the purest. König and Hühn found by their proposed method, in a sample of commercial sulphite wood cellulose, only 32·21 per cent. of "true cellulose"; this result is upheld against the criticism of Cross and Bevan on the ground that the sample still contained much lignin. The differences between the authors and Cross and Bevan turn on the question of a definition of chemically pure cellulose, as contrasted with the technically pure article. König and Braun, by the isolation of lignin, free from accompanying cellulose, but still retaining the structural form of the cell membrane, have proved that the lignocellulose of wood is not a chemical combination, but a mixture of two mutually interpenetrating colloids. J. F. B.

Determination of Bleaching Quality of Sulphite Pulp. E. Richter. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 13, 233-236.)—The author has previously shown that the Hempel-Seidel nitric acid figure of pulp can be employed to determine the percentage of non-cellulose in pulp, jute, or wood, by previously extracting the pulp with ether, alcohol, and water. The nitric acid figure gives values proportional to the bleach test, and an attempt has been made by a colorimetric method to get a value in per cent. of bleach powder. Sulphuric acid which has been employed by Klason (*Papier Zeit.*, **35**, 3781) as the basis of a colorimetric method for this purpose is unsatisfactory, since this acid attacks too much pure cellulose. The author recommends the following procedure: 5 grms. of air-dried pulp are broken up in thin pieces and placed in a wide-mouthed bottle with 100 c.c. of 13 per cent. nitric acid, well shaken, and kept in the dark for an hour. A standard pulp of known bleaching quality is put on alongside. At the end of the hour the mass is filtered through a small piece of pure absorbent cotton on a dry funnel, and 25 c.c. of the filtrate from each transferred into small bottles. From a burette water is added to the darker one until the shade of both solutions is the same. The colours can then be accurately compared in Eggertz carbon tubes. The bleaching quality of the pulp to be tested is calculated by simple proportion from the amount of bleaching powder that is known to be required for the standard. A strongly digested pulp will not always yield much colour to the acid in one hour, but placing the water-bottles in a water-bath at 40° C. and shaking will bring out the colour in about fifteen minutes, and the comparison can then be proceeded with. H. F. E. H.

Detection and Determination of Cyanogen and Hydrogen Cyanide. F. H. Rhodes. (*J. Ind. and Eng. Chem.*, 1912, 4, 652-655.)—The author has examined, from the quantitative point of view, the observation of Wallis (*Ann. Chem.*, 1906, **345**, 353), that while hydrogen cyanide in the presence of cyanogen is quantitatively precipitated when passed through acidified silver nitrate solution, cyanogen may be removed from solution in this reagent by a current of air. He finds that hydrogen cyanide and cyanogen may be determined accurately in the presence of each other, the former being precipitated as silver cyanide in acid silver nitrate solution (10 per cent. with 1 drop of dilute nitric acid), while the cyanogen which passes on may be detected and estimated colorimetrically by collecting it in $\frac{N}{2}$ caustic potash solution, adding ferrous sulphate and 1 drop of ferric chloride, and then, after fifteen minutes, acidifying with dilute sulphuric acid. In this manner as little as 0·3 c.c. of cyanogen can be detected in a mixture of the two gases, and in the presence of large volumes of air. Carbonic acid does not interfere unless present in such quantity as to form potassium carbonate with the whole of the $\frac{N}{2}$ caustic potash employed. If the quantity of hydrogen cyanide is very small, the trace of silver cyanide obtained can be filtered off and sublimed with a trace of iodine in a tube, when as little as 0·1 mgrm. of silver cyanide, equivalent to 0·02 mgrm. of original hydrogen cyanide, can be detected as a sublimate of cyanogen iodide.

H. F. E. H.

Estimation of Dextrose in the Presence of other Optically Active Substances by Bertrand's Method. M. Rosenblatt. (*Biochem. Zeitsch.*, 1912, **43**, 478-480.)—When it is desired to estimate dextrose in a solution containing, in addition, such substances as peptones, amino-acids, etc., by the polarimetric method, it is necessary to remove these substances previously by means of mercuric acetate and phosphotungstic acid (*Cf.* ANALYST, 1912, 145). Such is not the case, however, when the sugar is estimated by Bertrand's method (ANALYST, 1908, **33**, 476), as the results obtained are not affected to any appreciable extent by the presence, in the sugar solution, of glycin, alanin, leucin, tyrosin, asparagin, betaine, glutamin, urea, and peptone. This particularly applies to the estimation of dextrose in urine, for which purpose the polarimetric method is unsuitable, owing to the small quantity of sugar present. Urea and peptone have the most influence on the results obtained by Bertrand's method, the amount of sugar found being too low by from 1·6 to 4·4 per cent. of the quantity actually present. It is pointed out that the ferric sulphate solution used in Bertrand's method should contain 50 grms. of ferric sulphate and 200 grms. (not c.c.) of concentrated sulphuric acid per litre.

W. P. S.

Diphenylamine Test for Gun-Cotton. C. Kullgren. (*Zeitsch. f. Schiess- u. Sprengstoffwesen,* 1912, **7**, 153.)—Guttmann's test consists in maintaining the dried nitrocellulose at 80° C. in a tube in which is suspended a strip of diphenylamine test-paper, and taking as a measure of the stability of the sample the time required for the evolution of oxides of nitrogen sufficient to produce a blue tint on the paper. This investigation is to determine whether the acid substances producing this coloration exist ready-formed in the gun-cotton, or whether they are produced during the heating. The experimental material was a factory gun-cotton containing 12·6 per cent. of nitrogen, and dried at 40° to 45° C. for sixteen hours. Free acid in gun-cotton may be either the remains of nitrating acid not removed in manufacture, or the result of slow decomposition at ordinary temperatures; it was found that washing before the test had no effect if the sample was dried, because the acid is in any case driven off by drying at 45° C. This was supported by the observation that gun-cotton moistened with strong mineral acids recovered its test with sufficient drying; that drying at 45° C. gave rise to a much higher test than drying over phosphorous pentoxide or sulphuric acid at room temperature; and that a gun-cotton, insufficiently stabilised by boiling, gave a much higher test after being dried at 45° C. for two days than it did after standing for fourteen days at ordinary temperature, and drying for three hours at 45° C. The test is thus dependent, not on the free acid present in the gun-cotton, but on that produced during the test (*cf.* Robertson and Smart, *Zeitsch. f. Schiess- u. Sprengstoffwesen*, 1910, **5**, 185, 208). The danger of obtaining fallacious tests due to traces of unstable bodies may be avoided by prolonging the time of drying, and taking tests at intervals.

The decomposition of nitrocellulose is stated to take place in at least two stages, the velocity with which the intermediate body or bodies cause the evolution of oxides of nitrogen being increased more rapidly by rise of temperature than their velocity of formation. The test would then depend, not on the total amount of unstable constituents, but on the amount of intermediate product. Prolonged drying at 45° C. would diminish the amount of intermediate product, with a high test as the result; standing at ordinary temperatures would increase it, and a lower test would be produced. O. E. M.

Gasometric Estimation of Ether and Chloroform Vapours in Air. M. Koehmann and W. Streeker. (*Biochem. Zeitsch.*, 1912, **43**, 410-417.)—The quantity of ether or chloroform vapour in air may be estimated by shaking the gaseous mixture with alcohol and noting the decrease in volume of the former. The most suitable apparatus to use is a Bunte burette, and 96 per cent. alcohol is employed for the absorption. After the vapours have been absorbed, the alcohol vapour remaining in the burette is removed by means of water saturated with air. The necessary allowance is made for the vapour-pressure of water in calculating the decrease of volume due to the absorption of the ether or chloroform vapour, and full details as regards manipulation are given in the original paper. It may be mentioned that, in some preliminary experiments, olive oil and paraffin were used to absorb the ether and chloroform vapours, but the results obtained were unsatisfactory, owing to the formation of numerous small bubbles of air in the oil during the shaking part of the process. W. P. S.

Determination of Humus in Hawaiian Soil. W. P. Kelley and W. McGeorge. (*J. Ind. and Eng. Chem.*, 1912, **4**, 664-667.)—The author defines humus as the organic matter of soils soluble in 4 per cent. ammonia after removing calcium and magnesium with hydrochloric acid, and in the analyses given varies from 2·6 to 18·3 per cent., when estimated by the method described in the paper. A large number of soils were tested for humus content by the following methods, and the results tabulated: Hilgard, Mooers-Hampton, Houston-McBride (official), Cameron-Breazeale, and Rather. In addition the author describes a modification of his own, involving the filtration, under diminished pressure, of the humus solution through a

Pasteur-Chamberland filter, in order to separate the clay, the presence of which in soils containing much of it constitutes the most serious difficulty in the way of accurate determinations (*cf.* ANALYST, 1912, 466). When the first portion of the solution has passed the filter, 4 per cent. ammonia is drawn through as long as any colour is shown, and the filtrate and washings are then evaporated to dryness. The clay is washed out of the inside of the candle with a test-tube brush between each washing. The candle, with a short portion of its neck protruding, is suspended in a bell-jar from which the air is exhausted, the filtrate collecting in a jar beneath, while the solution to be filtered is poured into the candle down its centre from outside the bell-jar. Higher results are obtained by this process than by any of the others, excepting the official and Rather methods, neither of which is to be relied on for Hawaiian soils, as the majority of these are rich in very finely divided clay. The cause of the inaccuracy of these two last-mentioned methods becomes clear when the ash is determined in the humus obtained by evaporating its solutions to dryness, the value rising to as high a figure as 33 and 25 per cent. respectively, as against 0·6, 0·4, and 0·3 per cent. by the other methods. The large amount of clay unremoved from the humus solution accounts for the higher values. H. F. E. H.

Estimation of Methyl Alcohol in Admixture with Alcohol, and Especially in Spirits. W. Koenig. (*Chem. Zeit.*, 1912, **36**, 1025-1028.)—The following modification of the bichromate method of Thorpe and Holmes (ANALYST, 1904, **29**, 113) is recommended as the most generally applicable. The oxidation is effected in a litre flask (previously cleansed with boiling potassium permanganate solution and dilute sulphuric acid), and the carbon dioxide is conducted through drying-tubes, one containing sulphuric acid on pumice-stone, and the other calcium chloride, and is absorbed in two weighed U-tubes containing soda-lime, whilst beyond these are two more guard-tubes containing soda-lime, and calcium chloride. The oxidation flask is closed with a rubber cork with two openings, through one of which passes a dropping funnel, which can be closed at the top by a soda-lime tube, whilst the other opening receives a reflux condenser connected at its other end with the series of absorption tubes. After a current of air (freed from carbon dioxide) has been swept through the apparatus, the mixture of alcohol and methyl alcohol (5 to 10 c.c.) is introduced into the flask, and the dropping-funnel charged with a solution of 30 grms. of potassium bichromate in 500 c.c. of water and 50 c.c. of sulphuric acid (sp. gr. 1·84), this liquid having been first boiled for fifteen minutes and then cooled to 5° C. After this has also been introduced, the oxidation flask is shaken and allowed to stand for four hours, and its contents then slowly heated and boiled for one hour. Air is next drawn through the apparatus for about forty-five minutes, at first slowly, and then more rapidly, and the absorption tubes are then weighed. The weight of carbon dioxide multiplied by the factor 0·728 gives the amount of methyl alcohol. In test experiments with pure ethyl alcohol the amount of carbon dioxide corresponded to an average proportion of 0·50 per cent. of methyl alcohol—a result that agreed well with the observation of Thorpe and Holmes. The alcohol distilled from spirits behaved in the same way as pure alcohol, and in the case of test-mixtures containing from 4 to 15 per cent. of methyl alcohol the amounts found agreed within ±0·19 per cent. of the theoretical quantities. C. A. M.

Rapid Methods for determining Phenol. I. V. Redman and E. O. Rhodes. (*J. Ind. and Eng. Chem.*, 1912, **4**, 655-659.)—The authors compare the bromide-bromate method of Koppeschaar (*Zeitsch. anal. Chem.*, 1876, **15**, 233) with the hypobromite method worked out by Lloyd (*J. Amer. Chem. Soc.*, 1905, **27**, 16). A special shaking machine was employed, and it was found that, using either method, phenol determinations could be made within an error of 0·3 per cent. with one minute of continuous shaking after the solution containing the bromide was added, in place of the usual thirty minutes. It is important that the phenol solution be diluted until approximately $\frac{N}{100}$, and under these conditions the precipitate obtained by either method was white and flocculent, and showed no trace of red tetrabromo-phenoquinone or yellow tribromophenol bromide. The phenol solution employed must be acid after the bromine is added; if alkaline, an error arises, which increases as the concentration of the phenol present diminishes and the reaction period increases. The error introduced by shaking the solution for only one minute after the potassium iodide solution is added before titrating back with thiosulphate is 0·5 per cent. Three minutes shaking eliminates this, and further shaking has no effect. The potassium bromide-bromate solution (3·5 grms. bromate and 55 grms. of bromide to the litre) has the advantages over the $\frac{N}{10}$ hypobromite solution that it is permanent in strength and is free from unpleasant odour and free bromine, and only requires standardising once in three months, whereas the hypobromite solution weakened one-third per cent. every twenty-four hours for the first few days after being made.

H. F. E. H.

"Acid Resins" from Petroleum Residues. F. Schwarz. (*Chem. Rev. Fett- u. Harz.-Ind.*, 1912, **19**, 210-212.)—The acid residues obtained in refining the lighter distillation products of petroleum with sulphuric acid yield when diluted with water resinous substances which are used in the manufacture of varnishes, etc., while the acid is regenerated. In the cases of lubricating oils, however, the mineral acid cannot be separated in this way, and the residues are therefore neutralised with lime and used as asphaltum substitutes. As these products from the so-called "acid resins" are inferior to other asphalt materials, it is desirable to distinguish them from the more valuable distillation residues of petroleum. Samples of the former examined by the author yielded aqueous extracts which were strongly alkaline, owing to the presence of free lime. The presence of sulphonated acids was indicated by extracting the sample with petroleum spirit, evaporating the extract, igniting the residue, and testing the ash for sulphuric acid. In each case a small amount was found. For the separation of the organic acids, 5 grms. of each sample were thoroughly extracted in a basin on the water-bath with a mixture of absolute alcohol and a little hydrochloric acid (sp. gr. 1·19), and the extracts united, cooled to the ordinary temperature, and decanted from the residue. The clear brown liquid was then diluted with an equal volume of water, rendered alkaline with sodium hydroxide, and treated with petroleum spirit to separate unsaponifiable matters, after which the alcohol was evaporated from the alcoholic solution, and the residual aqueous solution shaken with ether and a little hydrochloric acid. On washing the ethereal layer with water, part of the organic acids was taken up by the water—this being an indication of the presence of sulpho-acids. The hydrochloric acid was therefore eliminated by washing the ethereal solution with a concentrated solution of sodium chloride, and the ether then evaporated. The residual acids were brown and resinous. They dissolved in water, forming a clear brown solution, but were only sparingly soluble in petroleum spirit and benzene. When neutralised and ignited they left an ash containing sulphuric acid. The amounts of sulphonated acids thus separated from three commercial samples were 7·5, 0·86, and 3·5 per cent. respectively. A distillation residue from petroleum treated in the same way yielded only an insignificant quantity of a yellow, resin-like mass which was insoluble in water. C. A. M.

General Method for the Estimation of Tartaric Acid. A. Kling and D. Florentin. (*Bull. Soc. Chim.*, 1912, **11**, 886-895.)—The addition of ammonium citrate prevents the interfering action of iron and aluminium salts on the estimation of tartaric acid as calcium racemate (*cf.* ANALYST, 1910, **35**, 323), even when the quantity of iron oxide or alumina present in certain pharmaceutical preparations amounts to 50 per cent. of the amount of tartaric acid. The effect of copper and antimony salts on the estimation is also prevented by the addition of ammonium citrate. The quantity of citrate added should be about ten times the weight of iron oxide or alumina present. When the proportion of alumina is very large, it is better to remove it by precipitation in the form of ammonium-alum. For this purpose the tartaric acid solution is treated with ammonium sulphate, a small quantity of hydrochloric acid, and alcohol; the alum crystals are collected on a filter, washed with alcohol, and the tartaric acid is estimated in the filtrate. Large quantities of iron are removed as sulphide in acetic acid solution. W. P. S.

INORGANIC ANALYSIS.

Titration of Copper Salts with Titanous Chloride. L. Moser. (*Chem. Zeit.*, 1912, **36**, 1126-1127.)—In the titration of copper salts by means of titanous chloride (see Rhead, *J. Chem. Soc.*, 1906, **89**, 1491) the use of thiocyanate and of a ferrous salt as indicator may be dispensed with. The reaction only proceeds quantitatively in strongly acid solutions, and hydrochloric acid is preferred because cupric solutions containing much hydrochloric acid possess a high colour, and the disappearance of this colour marks the end-point of the titration. For the same reason the author makes use of less dilute solutions than Rhead, a $\frac{N}{10}$ solution of titanous chloride being employed, and the copper solution concentrated to approximately $\frac{N}{5}$ strength and mixed with its own bulk of recently boiled hydrochloric acid (1 : 1) before the titration, which must be conducted in a vessel through which a current of carbon dioxide passes. The method is accurate to within 0·05 c.c. of the standard titanous chloride. G. C. J.

Detection and Estimation of Minute Quantities of Fluorine in Waters, Minerals, and Living Tissues. A. Gautier and P. Clausmann. (*Bull. Soc. Chim.*, 1912, 11, 872-884.)—The fluorine is first concentrated by rendering the water slightly alkaline, adding about 0·4 grm. of sodium sulphate together with a slight excess of barium chloride, and evaporating the whole to dryness. The residue is treated with a quantity of water sufficient to dissolve the soluble salts, an equal volume of 96 per cent. alcohol is added, the precipitate is separated by centrifugal action, and washed with alcohol until free from chlorides. In the case of minerals, those which are entirely attacked by sulphuric acid may be tested directly, whilst others are fused with sodium carbonate, dissolved in water, the silica is separated by means of ammonium carbonate, and the solution concentrated. Organic tissues are ignited in the presence of lime, the residue is dissolved in dilute hydrochloric acid, and the solution treated as in the case of water. When a mineral water containing much sulphate is under examination, magnesium chloride, ammonium phosphate, and ammonia may be added, and the precipitate produced employed for the test.

The barium, or other precipitate, obtained as described above, is now heated with concentrated sulphuric acid in a specially constructed gold crucible in such a way that the evolved gases are absorbed by moistened potassium hydroxide contained in a device which covers the crucible. At the end of two hours the potassium hydroxide is dissolved in water, the solution is brought just to boiling, nearly neutralised with hydrochloric acid, and ammonium chloride solution is added until the mixture, while still hot, is neutral to phenolphthaleïn. About 4 grms. of ammonium carbonate are then added, the solution is evaporated to dryness, the residue is dissolved in water, filtered, sodium sulphate is added to the filtrate, which is now heated to boiling, and precipitated with a slight excess of barium nitrate. The mixture is again evaporated to dryness, water is added, together with an equal volume of alcohol, the precipitate is separated by centrifugal action, washed with 65 per cent. alcohol until the last traces of nitrate and chloride are removed, then dried and weighed. A portion of this precipitate may be tested for the presence of fluorine by heating it with sulphuric acid and noting whether the vapours will etch glass. A lead crucible is used in this test, a varnished glass-cover having a small unvarnished portion being employed to cover the crucible. The quantity of fluorine present is estimated in the remaining part of the weighed precipitate by heating the latter with sulphuric acid in a platinum crucible, and absorbing the vapours by powdered lead-glass moistened with water. The lead-glass is then dried, and washed with boiling 85 per cent. alcohol; the lead fluoride is dissolved by washing the lead-glass with a saturated potassium chlorate solution diluted with four times its volume of water, and, after the addition of a small quantity of gelatin solution, the lead is estimated colorimetrically as sulphide, its quantity being a measure of the amount of fluorine present. It is stated that the method will estimate accurately as little as 0·1 mgrm. of fluorine in 1 litre of water, or 100 grms. of vegetable or animal tissue, whilst the qualitative test described will detect as little as 0·001 mgrm. of fluorine.

W. P. S.

Sampling Gold Bullion. F. P. Dewey. (*Eighth Inter. Cong. App. Chem.*, 1912, Vol. I, 155-181.)—In many cases a chip or drill sample may be satisfactory, in others, drillings will give a more representative sample than chips. But with bullion concerning which nothing is known the sample must be remelted, properly mixed, and dipped. In many cases, particularly of cyanide bullion, the composition of the metal interferes with the actual assaying, and the bullion must be refined before the gold can be determined accurately. The paper includes the results of hundreds of assays which support the above conclusions. The degree of divergence between duplicates, when remelting is dispensed with, may be as much as 1 per cent.

G. C. J.

Estimation of Hydrogen and Methane in Gas Mixtures. W. Hempel. (*Zeitsch. angew. Chem.*, 1912, **25**, 1841-1845.)—The temperature obtainable by means of an ordinary Bunsen burner is insufficient to ensure complete combustion of methane in a Drehschmidt platinum capillary. The use of a small blowpipe flame is essential. The Drehschmidt platinum capillary may be replaced by a capillary of transparent quartz, into which is pushed a short length of platinum wire of such diameter that the wire almost fills the tube. Such a capillary has the advantage that there is no risk of propagation of flame from the heated portion of the capillary to an explosive mixture contained in the gas burette to which it is connected.

When using the Winkler-Dennis combustion pipette there is often difficulty in burning the last of the methane, so that the operation extends over a considerable period, during which such an amount of mercury may be oxidised as to give rise to serious error. This source of error may be avoided by enclosing the electrically heated spiral in a quartz glass tube, which becomes the outer limb of the capillary U-tube of a simple mercury pipette. The gas is passed slowly to and fro between the burette and pipette over the heated spiral. The quartz tube is connected at either end by short pieces of rubber tubing to the tubes of soft glass, through which pass the stout platinum wires conveying the current to the spiral. A detailed drawing of a convenient arrangement accompanies the paper.

Fractional combustion of hydrogen by means of palladium asbestos is successful only if the passage of gas through the capillary be slow and the temperature of 400° C. not exceeded. A convenient way of controlling the temperature is to let the capillary containing the palladium asbestos lie horizontally in a groove cut in a massive block of brass, which is heated by a flame. Close to the groove in the block is bored a vertical hole which accommodates a thermometer or a tube of mercury, the boiling of which shows that a suitable temperature has been reached. The author uses a capillary of 1 to 1½ mm. diameter, filled for 10 mm. of its length with palladium asbestos, and it is found that the speed of the gas through such a capillary should not much exceed 10 c.c. per minute.

The use of a palladium sol, together with a soluble picrate as an absorbent for hydrogen, as suggested by Paal and Hartmann (ANALYST, 1910, **35**, 139), gives excellent results, but, as originally described, the method is rendered tedious by the persistent frothing of the liquid. Moreover, the absorptive capacity of the reagent falls off somewhat rapidly on storage, even in the dark. The froth is instantly destroyed by the introduction of 5 drops of alcohol through the capillary, but alcohol makes the reagent useless for further determinations. As the reagent is unaffected by mercury, the most convenient procedure is to use but a small quantity of it confined over mercury. The author treats 0·3 grm. or less of Kalle's colloidal palladium with 5 c.c. of water, and after twenty-four hours the palladium sol is mixed with 10 c.c. of a saturated solution of sodium picrate and charged into a mercury pipette. The quantity named has an absorptive capacity of about 455 c.c. of hydrogen, and can be depended on to absorb a quarter of that amount quantitatively and quickly from a gas mixture. Alcohol is used to destroy foam, and the contents of the pipette are then transferred to the palladium residue bottle, and the pipette is recharged after being washed free from traces of alcohol. G. C. J.

Methods for the Estimation of Iodides. W. Schirmer. (*Arch. Pharm.*, 1912, **250**, 448-451.)—The ferric chloride, nitrite, and iodate methods for the estimation of iodides were found to yield the most trustworthy results under the following conditions:

Ferric Chloride Method.—About 0·4 grm. of the iodide is dissolved in not more than 20 c.c. of water, and the mixture is treated with 5 grms. of ferric chloride solution; after the lapse of one hour the mixture is diluted with 120 c.c. of water, 10 c.c. of phosphoric acid (25 per cent.) are added, the liberated iodine is dissolved by the addition of 0·5 grm. of potassium iodide, and titrated with thiosulphate solution. One c.c. of $\frac{N}{10}$ thiosulphate corresponds with 0·0166 grm. of potassium iodide.

Nitrite Method.—In a stoppered flask 0·5 grm. of the iodide is treated with 50 c.c. of water, 10 c.c. of dilute sulphuric acid, and 10 c.c. of a 1 per cent. sodium nitrite solution. At the end of two minutes, 1 grm. of urea is added, the contents of the flask are shaken for five minutes, and, after the separated iodine has been dissolved by the addition of 0·5 grm. of potassium iodide, the solution is titrated with thiosulphate solution.

Iodate Method.—Twenty c.c. of a 1 per cent. potassium iodate solution, 5 c.c. of dilute sulphuric acid, and 50 c.c. of water are placed in a stoppered flask, and 0·4 grm. of the iodide, dissolved in 10 c.c. of water, are introduced. After the lapse of about two minutes 5 grms. of borax are added, and at the end of a further five minutes the liberated iodine is dissolved by the addition of about 1 grm. of potassium iodide, and titrated with thiosulphate solution. W. P. S.

Applications of the Method of Precipitating Metals as Iodides, etc., from Sulphuric Acid Solution. G. Bressanin. (*Gazz. Chim. Ital.*, 1912, **42**, 97-101.)—

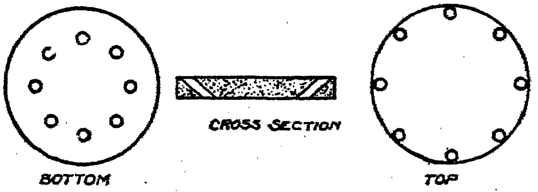
Estimation of Tin.—Solutions of tin in sulphuric acid (sp. gr. 1·52) yield a precipitate on treatment with potassium iodide in an analogous manner to arsenic and antimony (ANALYST, 1912, 206). The reaction, which is given by both stannous and stannic salts, is much more sensitive than the reaction of stannous salts with mercuric chloride. The tin precipitate is bright yellow, and resembles arsenic iodide, but is readily soluble in hydrochloric acid, whereas arsenic iodide is practically insoluble. It differs from antimony iodide in colour, and in being more soluble in hydrochloric acid. In order to obtain quantitative results it is necessary that the tin should be in the higher form of oxidation, since, unlike arsenic and antimony, which are precipitated as iodides, stannic compounds are precipitated as stannic iodate, whilst stannous precipitates are unstable and undergo continuous oxidation. With this precaution of preliminary oxidation of the tin, arsenic and antimony may be estimated in the presence of that metal. The stannic precipitate, when dissolved in dilute tartaric acid solution, gives a solution which, when neutralised with sodium bicarbonates, does not absorb iodine.

Estimation of Arsenic in Commercial Copper.—The proportion of arsenic in commercial copper is so small that a large amount of the copper is required for the estimation; and since the copper salts on treatment with potassium iodide liberate an atom of iodine for each atom of copper, the total quantity of iodine thus set free is so great that it cannot be eliminated by a single washing with a solution of potassium iodide in sulphuric acid. The process must therefore be modified as follows: The finely divided copper (0·5 grm.) is dissolved in 5 c.c. of *aqua regia*, the solution evaporated to dryness, and the residue, after removal of all nitric acid, dissolved in about 250 c.c. of hot sulphuric acid (sp. gr. 1·52). On cooling the solution, and adding 25 c.c. of a 30 per cent. solution of potassium iodide, the copper and arsenic are precipitated together as iodides. The precipitate is allowed to stand for about twenty minutes, after which it is collected on an asbestos filter, and washed with the aid of a filter pump with 30 to 40 c.c. of sulphuric acid containing 5 per cent. of 30 per cent. potassium iodide solution, and the arsenic iodide dissolved off the filter by means of a dilute solution of sulphurous acid, which also reduces the remaining iodine. If the filter bed is sufficiently close none of the copper iodide will pass into the filtrate, which should remain perfectly limpid. The filtrate and washings are treated with sufficient iodine solution to give a distinct blue coloration with starch paste, then rendered alkaline with sodium bicarbonate, and the titration completed with iodine solution. The arsenic is calculated from the amount of iodine used in this second period of the titration. C. A. M.

Colorimetric Determination of Iron in Lead and its Oxides. J. A. Schaeffer. (*J. Ind. and Eng. Chem.*, 1912, 4, 659-660.)—The method is substantially that of Thomson (*J. Chem. Soc.*, 1885, 47, 493), with the difference that greater care is taken to secure similarity in the solutions to be compared. In the analysis of litharge or metallic lead, 1 grm. of the sample is covered with 15 c.c. of water, and enough nitric acid is added to convert all the lead to nitrate. The solution is diluted if basic nitrate separates, is boiled for several minutes to peroxidise iron, and is then neutralised with ammonia, and again rendered faintly acid by addition of a measured amount of dilute nitric acid. It is then transferred to a 100 c.c. Nessler glass, 15 c.c. of thiocyanate (5 per cent.) is added, and the mixture diluted to the mark, and its colour matched by adding a standard solution of ferric ammonium sulphate from a burette to a cylinder containing 15 c.c. of thiocyanate and as much nitric acid as was added to the test solution after neutralisation.

In the analysis of red lead, the initial treatment is the same, but when the solvent action of the nitric acid slackens, 10 c.c. of hydrogen peroxide (2 : 7) is added, and the solution boiled for one minute. The solution is neutralised, acidified again by addition of a measured quantity of dilute nitric acid, and cleared, if necessary, by addition of a few drops of hydrogen peroxide solution. To the comparison cylinder 10 c.c. of hydrogen peroxide solution (2 : 7) as well as a measured quantity of nitric acid and thiocyanate are added. Quantities of iron up to 0·03 per cent. can be estimated with an error not exceeding 0·001 per cent., and the small percentage of copper present in lead and its oxides, such as are used in the manufacture of accumulators and high-grade lead-glass, does not interfere. G. C. J.

Determination of Carbon in Steel by Direct Combustion in the Newest Form of Shimer Crucible, with the Aid of a Perforated Clay Disc. F. O. Kichline. (*J. Ind. and Eng. Chem.*, 1912, 4, 683-684.)—Description of improvements in the platinum crucible with water-cooled brass stopper previously described by P. W. Shimer, and of modifications in the manner of using it for carbon determinations. The crucible is now made of slightly larger diameter, so as to accommodate a No. 00 Berlin crucible instead of a No. 000. This allows the use of 2·727 grms. of drillings (1 mgrm. CO_2 then $=0·01$ per cent. C.), instead of only half this charge. The drillings are placed on a bed of sand, of which enough is taken to half fill the crucible, and are now covered by a perforated disc, like that shown in the illustration, instead of with asbestos as formerly. Allowing for these modifications, the improved apparatus is used in the manner already described. The discs are best made of China clay, which is mixed with water to the proper consistency, moulded into shape, carefully dried, fired and annealed. With a little practice they can be constructed in any ordinary laboratory, and with care in use will last for 100 determinations like the porcelain liners. Compared with asbestos they save time and are cleaner in working. G. C. J.



CLAY DISC.

Technical Analysis of Spelter. E. J. Ericson. (*Eighth Inter. Cong. App. Chem.*, 1912, Vol. I, 183-186.)—The spelter (20 grms.) is covered with 100 c.c. water and 50 c.c. dilute (1 : 3) sulphuric acid, and allowed to stand overnight. The lead, cadmium, and undissolved zinc are then filtered off, washed with hot water, returned to the beaker, and dissolved in 10 c.c. of nitric acid. The solution is boiled until brown fumes cease to be evolved, and is then diluted and filtered from any trace of tin or antimony oxide. If these are absent, as is usually the case, the solution is diluted to at least 100 c.c., and 30 c.c. of ammonia and 5 to 10 grms. ammonium persulphate are added, and the solution boiled for five minutes. After ten minutes, the lead peroxide is filtered off on a double filter (11 cm.) and washed four times with hot 10 per cent. ammonia and five times with hot water. The precipitate is washed back into the beaker and treated with a measured volume of hydrogen peroxide solution of known strength until dissolved. After addition of 15 c.c. of dilute nitric acid (sp. gr. 1·2) and 75 to 100 c.c. water, the excess of hydrogen peroxide is determined by titration with permanganate. If 1 c.c. of the permanganate corresponds to 1 mgrm. of iron, it should theoretically correspond to 1·85 mgrm. of lead; but experience shows that the factor 1·92 should be employed. Using this factor, the results check well with results obtained by the gravimetric chromate method, and the method is recommended for spelter containing 1 per cent. or less lead.

The ammoniacal filtrate from the lead peroxide is boiled until nearly neutral and a white precipitate appears. About 40 c.c. of dilute (1 : 3) sulphuric acid is then added, and boiling continued for ten minutes. After diluting to 200 c.c., the solution is saturated with hydrogen sulphide, with occasional additions of water. The precipitate is collected on a double filter, washed a few times, and then redissolved on the filter with the least possible quantity of hot dilute hydrochloric acid. Any copper which may be present remains on the filter as sulphide, and may be ignited and weighed as oxide. The solution of cadmium (and zinc) chloride is nearly neutralised with ammonia, and about 8 grms. of trichloracetic acid (*cf.* Fox, *J. Chem. Soc.*, 1907, **91**, 964) dissolved in water is added—or more, if more is necessary—to dissolve the cadmium sulphide which separates on neutralisation. The solution is diluted to 200 c.c., and the cadmium reprecipitated by hydrogen sulphide. The cadmium sulphide is filtered off, and finally converted into sulphate or phosphate.

G. C. J.

Determination of Manganese by the Bismuthate Method. W. Blum. (*Eighth Inter. Cong. App. Chem.*, 1912, Vol. I, 61-85.)—As the result of exhaustive research in the U.S. Bureau of Standards, the conclusion is reached that the bismuthate method is more accurate than any other, even for the analysis of high-grade manganese ores. It is shown that the manganese value of permanganate solution is identical, whether calculated from a sodium oxalate titration by means of the theoretical factor 0·1640, or determined by means of pure anhydrous manganese sulphate. Statements to the contrary (*e.g.*, that of Brinton, ANALYST, 1911, **36**, 305; see also Hillebrand and Blum, *ibid.*, 1911, **36**, 430) are probably due to the difficulty of depriving manganese sulphate of traces of free sulphuric acid without at the same time decomposing some of the manganese sulphate. It is shown that prolonged heating to at least 440° C. is necessary to eliminate free sulphuric acid, whilst at temperatures above 525° C. there is risk of decomposition. The sodium oxalate method of standardisation is therefore recommended. $\frac{N}{10}$ permanganate, which should be used in the analysis of high grade manganese ores, is standardised by dissolving as much sodium oxalate as will require nearly 50 c.c. in 250 c.c. of water containing 5 c.c. of sulphuric acid, warming to 80° to 90° C., and titrating slowly. The final temperature should not be below 60° C. $\frac{N}{30}$ permanganate, such as is used for determining manganese in steel, is standardised in a similar manner, but a sharper end-point is obtained by restricting the initial volume to about 75 c.c.

Analyses are conducted as follows: To the cold manganese solution containing 20 to 40 per cent. of nitric acid (free from nitrous acid) in a bulk of 50 to 150 c.c., a slight excess of bismuthate (0·5 to 1 grm.) is added, and the mixture agitated for half a minute. The sides of the flask are washed down with 50 c.c. of 3 per cent. nitric acid, and the solution is filtered at once through asbestos, previously coated with bismuthate. About 100 c.c. of 3 per cent. nitric acid is used to wash the flask and filter. A measured amount of standard ferrous sulphate solution is added, and the excess titrated with permanganate. For quantities of manganese up to 0·05 grm. the method is accurate to 1 in 500. The experience of the Bureau shows that no other known method, gravimetric or volumetric, gives results for which a higher degree of accuracy can be claimed. With quantities of manganese as large as 0·1 grm., the method as described gives rise to errors of several units per cent.

It is shown that permanganate solutions preserve their strength when exposed to diffused light, if protected from reducing substances, and that $\frac{N}{10}$ solutions of ferrous sulphate are as stable as those of ferrous ammonium sulphate, the rate of oxidation under average conditions being about 1 per cent. in five days (see also Baskerville and Stevenson, ANALYST, 1911, **36**, 467). G. C. J.

Rational Analysis of Nitrate of Soda. W. S. Allen. (*Eighth Inter. Cong. App. Chem.*, 1912, Vol. I, 19-31.) — A protest is raised against the custom of estimating the content of sodium nitrate in commercial nitrate of soda by subtracting from 100 per cent. the sum of the water, insoluble matter, and chloride and sulphate expressed as sodium chloride and sulphate. The method invariably overestimates the sodium nitrate, the average error being about 1 per cent.; but it may reach 3 per cent. Not only do all samples contain soluble salts of calcium, magnesium, iron, and aluminium in small amount, but sodium perchlorate is occasionally present up to nearly 1 per cent., and the percentage of potassium nitrate may exceed 8 per cent. Though commercial nitrate of potash is more valuable than the sodium salt, its presence in admixture with the latter is a disadvantage to the nitric acid maker, as its nitrogen content is lower than that of nitrate of soda. It is held that the basis of sales should be the nitrate content as determined by a direct method. A modification of the Devarda method (*Zeitsch. anal. Chem.*, 1894, **33**, 113) is described, and the opinion expressed that it will be found simpler than the use of the nitrometer by those who are not thoroughly familiar with the use of that instrument. The new method consists in reducing the nitrate to ammonia in alkaline solution by means of Devarda metal, and driving the ammonia over into a measured volume of standard acid by means of a current of air, which bubbles through the reduced solution contained in a flask which is heated in a water-bath. Between the flask and the receiver is a spray-tap, consisting of four 1-inch bulbs blown on a tube about 8 inches long. The bulbs are filled with glass-wool, and drain back towards the reduction-flask. To prevent undue condensation in this spray-trap, and to hasten the distillation of the ammonia, the bulbed tube is surrounded with a lead jacket through which steam is passed. The air which is drawn through the apparatus is previously freed from carbon dioxide, and methyl red is preferred as indicator. G. C. J.

Effect of Ignition on the Solubility of Soil Phosphates. C. B. Lipman. (*J. Ind. and Eng. Chem.*, 1912, **4**, 663 664.)—Fraps (ANALYST, 1911, **36**, 371) showed that the ignition of phosphate minerals such as wavellite, dufrenite, etc., for ten minutes at a low red heat increased about ten times the solubility of the phosphoric acid in them in $\frac{N}{5}$ nitric acid, and rendered it almost entirely soluble in 12 per cent.

hydrochloric acid. The author has frequently noticed the reverse effect when soils were ignited, and below are recorded the figures obtainable when the phosphoric acid is estimated in soils of varying composition before and after ignition:

	Percentages of P_2O_5.				
	1.	2.	3.	4.	5.
Ignited ...	0·410	0·150	0·090	0·167	0·151
Not ignited ...	0·460	0·250	0·130	0·191	0·190

Theoretical considerations are discussed as to the possible causes of the loss noticed and the reason for the reverse effect mentioned above. H. F. E. H.

Action of Dimethyl-Glyoxime on Platinum Salts. M. Wunder and V. Thuringer. (*Ann. Chim. anal.*, 1912, **17**, 328-329.)—Platinum salts form an insoluble compound with dimethyl-glyoxime, but with much more difficulty and at a higher temperature than the corresponding palladium compound (ANALYST, 1912, 379). The platinum precipitate is brown, blue, or greenish-blue while moist, but assumes a bronze colour when dry. In experiments to render the reaction quantitative the precipitate, obtained in the same manner as with palladium (*loc. cit.*), was digested for several hours on the water-bath, then separated, dried, and ignited. From 0·0292 grm. of platinum there was obtained 0·0222 grm. Better results (*e.g.*, 0·266 grm.) were obtained by the use of an alcoholic solution of dimethyl-glyoxime, while by adding sodium citrate to augment the reducing action the figures were still better (*e.g.*, 0·0250 grm. from 0·0262 grm.). For each part of platinum one and a half parts of the reagent were used. The presence of ammonium chloride accelerated the formation of the precipitate, but did not lead to better results than those quoted. C. A. M.

Rare Earth Reactions in Non-Aqueous Solvents. O. L. Barnebey. (*J. Amer. Chem. Soc.*, 1912, **34**, 1174-1189.)—A study of the reactions of the common acids and bases with the yttrium group—neodymium, lanthanum, and cerium—using acetone as solvent. The reactions are of the same general order as those in aqueous solution, but the solubilities vary more widely. In some cases compounds which are readily soluble in water have been found to be nearly insoluble in acetone, so that the use of acetone as solvent, in combination with reagents used for the separation of the earths by fractional precipitation, promises to make such separations sharper and less tedious. The basic nitrate scheme of fractionation, using acetone as solvent, is very effective as a means of separating ceria from the other earths, but none of the other separations described are separations in the sense in which the term is usually understood by analysts. A separation of the yttrium group, using tartaric acid as precipitant, has been found very effective for fractional purposes. The serial order established is ytterbium (lutecium and neoytterbium), holmium, erbium, dysprosium, yttrium. Other separations described depend on the use of alkaloids as fractional precipitants in acetone solution. G. C. J.

Exact Determination of Sulphur in Pyrites. W. S. Allen and H. B. Bishop. (*Eighth Inter. Cong. App. Chem.*, 1912, Vol. I, 33-51.)—The method depends on oxidation and solution by means of a solution of bromine in carbon tetrachloride followed by nitric acid, reduction of ferric iron by means of aluminium powder, and precipitation in cold dilute solution by slow addition of barium chloride solution. Large quantities are taken for analysis, so that the barium sulphate to be weighed approximates 5 grms. The results are accurate to within 0·05 per cent. Under the prescribed conditions, the barium sulphate always contains barium chloride, but the amount of this is very nearly constant (0·0137 to 0·0150 grm.), and the results show that the error due to this cause is almost exactly compensated by the solubility of the barium sulphate.

The sample is not ground more finely than 80 mesh (*Cf.* Allen and Johnston, ANALYST, 1910, **35**, 332), and 1·3736 grms. is weighed out into a 300 c.c. beaker and covered with 10 c.c. of a mixture of 2 volumes bromine and 3 volumes carbon tetrachloride. Bromine alone is too violent, and its aqueous solution too weak. The mixture is shaken from time to time, and after fifteen minutes 15 c.c. of nitric acid is added, and the mixture left for a further fifteen minutes at room temperature, with occasional shaking. The beaker is then placed on an asbestos board on the steam-bath until all action has ceased and most of the bromine has volatilised. The clock glass covering the beaker is next raised somewhat by means of glass riders, and the contents of the beaker are evaporated to dryness by immersing it in the steam-bath up to its rim. Hydrochloric acid (10 c.c.) is added, and the solution again evaporated to dryness, and the residue dried for several hours at 100° C. to render silica insoluble. The residue is moistened with 4 c.c. of hydrochloric acid, and after five minutes 100 c.c. of hot water is added, and the mixture boiled for five minutes. It is allowed to cool for about five minutes, and then 0·2 to 0·3 grm. of aluminium powder is added, and the contents of the beaker agitated until the colour of ferric iron disappears. The solution is cooled to arrest the action of acid on aluminium, which might otherwise give rise to mechanical loss on the filter, and is filtered through a 12·5 cm. filter, which is washed nine times. The filtrate is diluted to about 1,600 c.c., and a further 6 c.c. of hydrochloric acid is added to it. About 125 c.c. of 5 per cent. barium chloride solution is added without stirring, and at a rate not exceeding 5 c.c. per minute. The paper includes description of an appliance for effecting this without attention. When all the barium chloride has run in, the contents of the beaker are stirred, and then allowed to stand for not less than two hours. The liquid is decanted through a 35 c.c. Gooch filter with a moderately thick asbestos mat, and the precipitate is transferred to the filter and washed six times with cold water. The crucible is then dried and heated for half an hour with the full flame of a Bunsen burner (or in an electric oven at 870° C.). The weight of the precipitate multiplied by ten gives the percentage of sulphur in the sample. Test analyses show that the method gives results of the accuracy stated, and that it is unaffected by the presence of such amounts of lead, copper, zinc, arsenic, lime, and silica, as may occur in pyrites. G. C. J.

APPARATUS, ETC.

Crystallo-Chemical Analysis: A New Method of Chemical Analysis. G. von Fedorow. (*Zeitsch. f. Krystallogr.*, 1912, **50**, 513-575; through *Nature*, 1912, **89**, 503-505.)—It is shown that there is a correct mode of setting up a crystal for descriptive purposes, and that this setting is arrived at independently of external "habit," being based on calculations of the "reticular density" (closeness of packing of the nodes, or "points," of the space-lattice or point system) along the planes of the principal faces. The setting which corresponds to primary faces of maximum reticular density is the only correct one on which comparisons should be made. A table including 10,000 crystalline substances, which have been goniometrically measured, has been prepared, arranged in regular progressive order, and calculated on the above lines as regards correct setting. If a few measurable crystals of any one of these substances be subjected to a short goniometrical investigation, occupying at most a few hours, and often only a few minutes, the substance of which the crystal is composed can then be identified by reference to the table. In short, provided a chemical substance has once had its crystals measured, it is possible to detect it at any time by merely making a few brief measurements so as to be able to calculate the elements—by a shortened process, partly graphical, which the author has perfected—and then searching the table for the substance therein recorded as possessing such constants. A number of test cases have been sent to the author, and he succeeded in identifying them correctly. The method has the advantage of not injuring the crystals, but fails in its simple form with cubic crystals, in which the angles are always the same; but a determination of refractive index is successful where that of symmetry, elements, and angles fail.

H. F. E. H.

Luminescence Analysis. O. Wolff. (*Chem. Zeit.*, 1912, **36**, 1039.)—Lehmann has devised a fluorescence microscope which is used with ultra-violet light and has a screen for the protection of the eye. By means of this instrument the author has found that the red fluorescent particles in crude potashes consist of potassium sulphide, whilst the particles showing a blue fluorescence consist of organic matter, mainly in combination with the mineral matter. In like manner the orange fluorescence of mercuric chloride is shown to be due to particles of mercurous chloride. The fluorescence of the latter disappears on heating. Chemically pure mercuric chloride shows no fluorescence, but in many of the preparations on the market traces of calomel may thus be detected, although they escape detection by chemical means. Resublimation of pure mercuric chloride in a tube causes the orange fluorescence to appear, owing to the formation of traces of calomel. Another direction in which this microscope has proved useful is in the examination of anthracene, which when pure shows a blue fluorescence, but when impure fluoresces green, owing to the presence of a substance with a yellow fluorescence.

C. A. M.

New Type of Inorganic Filter for Laboratory Purposes. M. A. Williamson and P. A. Boeck. (*J. Ind. and Eng. Chem.*, 1912, **4**, 672-677.)—The material of the new filter is "alundum" (*cf.* Boeck, ANALYST, 1912, 288), a porous ware already in use for the construction of extraction thimbles and filter plates (ANALYST, 1912, 109). When given the form of an ordinary crucible, this ware was not successful in replacing the Gooch crucible with asbestos mat, because the upper portion of the crucible absorbed strong saline solutions, and the time required to wash the salt out from those portions of the crucible above the level where the suction was operative was so great as to outweigh any advantages the crucible might otherwise have possessed. Filtering-cones have now been constructed which overcome this difficulty, and which serve as combined filter and crucible. The use of the cone as filter is shown in Fig. 1. A funnel a little larger than the cone is equipped with a piece of soft rubber tubing about 1½ inches long and 1½ inches in diameter (the cones are 1¾ inches in diameter and 1¼ inches high), and the cone finds a seating on the moistened rubber. The filter and any precipitate it may contain can be washed perfectly in two or three minutes, and, after drying, can be ignited. The material is unaffected by sulphuric, nitric, or hydrochloric acid. It is somewhat hygroscopic, a cone of the dimensions given picking up about 1 mgrm. on exposure to the air of the laboratory for one hour, but there is no further gain in weight in three hours. Among the test numbers given, those for barium sulphate are all low—from 0·1329 to 0·1331 found against 0·1349 required by theory and found by ordinary filtration using a paper. Since parallel experiments in which a paper was placed inside the cones also gave 0·1330, the low results appear to be due not to penetration of the filter by the finely divided precipitate, but to the solubility of the precipitate in the washing water, of which there is a tendency to use large quantities when a filter operates very quickly. Fig. 2 shows how these cones may be freed from any soluble salts suspected to lie within their pores. The illustration actually figures a crucible-shaped filter, of the type which was abandoned owing to its tendency to absorb salts; but a similar arrangement serves to clean a cone, as to the cleanliness of which there may be a doubt. The cones are made by the Norton Company, Worcester, Mass., U.S.A.

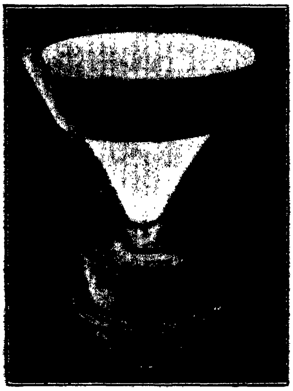


FIG. 1.

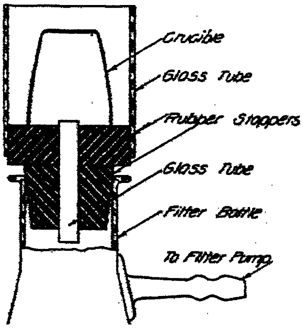
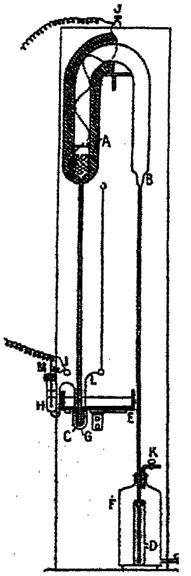


FIG. 2.

G. C. J.



Apparatus for Rapid Distillation of Mercury in Vacuo. L. Dunoyer. (*Compt. rend.*, 1912, **154**, 1344-1346.)—The apparatus consists of a bent glass barometer tube, *A B*, of 4 to 5 cm. diameter and 35 to 40 cm. high. The limb *A* terminates in a tube about 10 mm. in diameter, which dips into a small beaker, *C*, fixed to the tube. This beaker hangs in a tube, *G*, which is cemented into the bottom of the rectangular glass dish *E*, which can accommodate some 20 kgrms. of mercury. The end *B* of the barometer-tube terminates in a tube about 120 cm. long and not more than 3 mm. in diameter. This narrow tube dips into the vessel *D*, which is provided with a side outlet, and is itself contained within the bottle *F*. The bottle *F* is evacuated by connecting the cock *K* to a water-pump. This causes the mercury to rise to the level shown in *A*, and the circuit is then completed through the electrical heating device, and the distillation commenced. The use of the vessels *C* and *D* make it possible to clean or empty *F* without interrupting the distillation. The heating device consists of a winding of chrome-nickel wire, extending from the constriction of the tube *A* to the highest point of the bend, separated from the glass by insulating material and externally covered with a thick layer of some poor conductor of heat. It is to be noted that only the small portion of mercury in *A* and the vapour above it are heated by the current, the bulk of the mercury in *E* remaining cool until drawn up into *A* to replace mercury which has evaporated.

The apparatus possesses a simple device for breaking the electrical circuit if, and when, the supply of mercury in *E* is exhausted or reduced to some assigned level. From the base of the vessel *H* proceeds a narrow S-tube, the other end of which dips into the mercury in *E*. *H* is closed by a wooden plug through which is screwed an electrode, *M*, of amalgamated copper tube. The height of this electrode is adjusted until its lower end corresponds to the level at which it is wished to arrest the further depletion of the mercury in *E*. Mercury is then poured into *H*, covered with a layer of petroleum, and the plug carrying the electrode is pushed into *H*, whilst closing the upper end of the tubular electrode with the thumb or a stopper. In this way mercury is forced into the S-bend, thus establishing electrical connection between the electrode and the mercury in *E*. The current passes from the terminal *I* through the electrode *M* and the mercury in *H*, the S-bend and *E*, and thence by the nickel wire *L*, which dips into the mercury in *E*, to the heating coil, the other terminal of which is at *J*. When the level of the mercury in *E* has sunk to the level of the lower end of the electrode *M*, the electrical circuit is automatically broken and the distillation is suspended. G. C. J.

Two New Pieces of Laboratory Apparatus. E. Berlin. (*Zentralbl. für Physiol.*, 1912, **26**, 219; through *Chem. Zentralbl.*, 1912, II, 889.)—The first apparatus is for the evolution of hydrochloric acid gas from its aqueous solution by the action of sulphuric acid. It consists of a round-bottomed flask, into the closed top of the neck of which are sealed two tubes, each terminating downward somewhat below the base of the neck inside the flask, and leading upward, through a stopcock, to a bulb carrying a funnel and a tube opening to the air; funnel and tube are simultaneously opened and closed by the plug of a stopcock passing through both. A delivery tube, and an emptying tube reaching the bottom of the main flask, are sealed into the neck halfway up, and are closed with stopcocks. The bulbs are charged with concentrated hydrochloric acid and concentrated sulphuric acid respectively.

The second apparatus is for the continuous extraction of a liquid, such as water, by a heavier liquid, such as chloroform. Into the neck of a round-bottomed flask with reflux condenser is sealed a tube with its mouth, directed downwards, a short distance below the cork. This tube passes down outside the neck to join a second tube, sealed through the base of the neck, and reaching the bottom of the flask. From the junction the tube is widened, and passes diagonally downwards to a smaller flask in which chloroform is boiled. Chloroform is placed in the larger flask until the mouth of the tube reaching to the bottom is well covered, and on this sufficient of the aqueous liquid to be extracted to cause the chloroform to overflow into the smaller flask. Chloroform, as vapour, passes up the side tube, is condensed, sinks in drops through the aqueous liquid, and flows over into the smaller flask. O. E. M.

Apparatus for the Estimation of Adulterants in Vegetable Powders, and Notes on Starch Granules. C. Hartwich and A. Wichmann. (*Arch. Pharm.*, 1912, **250**, 452-471.)—The apparatus consists of a microscope slide on which is engraved a square, the sides of which measure 1·5 cm.; this square is divided into 100 equal squares, and around its edges are placed strips of cover-glass, so that a shallow cell, 0·25 mm. in depth, is formed. The powder under examination is diluted with a known quantity of sugar, say in the proportion of 1 to 100 or 1 to 1,000, and a small quantity of the mixture is weighed out into the cell. For instance, if 0·05 grm. of the mixture (1 to 100) be taken, the cell will contain 0·0005 grm. of the original powder. A drop or so of water is then added to the cell, and, when the sugar has dissolved, a cover-glass is placed over the whole, and the slide is examined under the microscope, the foreign particles being counted. The apparatus is particularly suitable for estimating the amount of clove-stalks in powdered cloves, sandal-wood in saffron powder, starches in powders, etc. The author has also determined the weight of various starch grains by means of the apparatus, the weight of a single grain, after the starch had been dried at 100° C., being: *Oryza sativa*, 0·0000000000163 grm.; *Zea Mais*, 0·000000000729 grm.; *Maranta arundinacea*, 0·0000000063 grm.; *Triticum sativum*, 0·0000000000605 grm.; *Canna edulis*, 0·0000000502 grm.; *Solanum tuberosum*, 0·00000000644 grm. The specific gravities of the dry starches were found to be: *Canna edulis*, 1·5255; *Solanum tuberosum*, 1·5247; *Maranta arundinacea*, 1·5215; *Oryza sativa*, 1·5134; *Zea Mais*, 1·4809; *Triticum sativum*, 1·4696. W. P. S.

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REPORT.

Report of the Government Chemist upon the Work of the Government Laboratory for the Year ending March 31, 1912, with Appendices. (*Issued as a Parliamentary Paper*, pp. 1-27.)—The Report commences with a short recapitulation of the principal steps in the history of the Laboratory and its work, from the origin of the Laboratory in 1843 to the year 1911, when the Government Laboratory was separated from the Department of Customs and Excise, and constituted a new Department under the official title of the " Department of the Government Chemist." Changes in the method of staffing the Laboratory have also been made, and, whilst Revenue Officers still attend to that part of the work which pertains to the Revenue, chemists recruited from " the open market " are employed for non-Revenue work. The duties performed by the staff include analyses of samples taken by Revenue officers, of stores supplied to Government Departments, of dairy produce imported into this country, of samples referred by magistrates under the Food and Drugs Act, the Fertilisers and Feeding-Stuffs Acts, etc.

The total number of analyses and examinations made in the two branches of the Laboratory during the year was 195,170, compared with 186,044 for the preceding year. Including samples examined in provincial stations, the total number was 345,181.

For the purpose of checking dilution and adulteration 7,464 samples of beer were taken from retailers, and of this number 452 were found to have been diluted. None of the samples contained saccharine. Of 444 samples of herb beers and temperance beverages examined, 300 contained less than 2 per cent. of proof spirit; 107 contained less than 3 per cent.; 30 between 3 and 5 per cent.; 6 between 5 and 8 per cent.; and 1 sample 10·6 per cent. One thousand and forty-six samples of beer and brewing materials were examined for the presence of arsenic, but only 18 of these contained more than one-hundredth of a grain of arsenious oxide per gallon (or pound, in the case of solids). Of 122 samples of malt 5 exceeded the limit, the highest proportion found being one-fortieth of a grain per pound; 8 samples of wort and beer contained excessive quantities of arsenic—the highest quantity being one-twentieth of a grain per gallon. The highest quantity of arsenic found in 377 samples of other brewing materials was one-fiftieth of a grain per pound. In addition to the samples connected with brewing, 292 samples of mineral acids, chemicals, etc., were tested for arsenic. Attention is drawn to the fact that 19 samples sold as cider were found to be artificially prepared liquids, and to be free from apple-juice. A considerable proportion of the 56 samples of liqueur chocolates analysed was found to contain from 6 to 12 per cent. of proof spirit, and in two cases the quantity reached 25·0 and 28·8 per cent. respectively.

In connection with the Board of Agriculture and Fisheries, and the Department of Agriculture and Technical Instruction for Ireland, 3,228 samples were examined, including 70 samples of milk, 63 of condensed milk, 71 of cream, and 1,309 of butter. Only 11 of the samples of imported butter contained excessive quantities of water; 396 contained boric preservative, and 180 added colouring matter. The samples of imported milk, cream, and butter are always tested for the presence of preservatives

The whole milk imported in churns was usually free from preservatives, but 3 samples contained boric acid and 1 hydrogen peroxide. Boric acid is generally present in imported cream, and its use in past years has been supplemented by the addition of salicylic acid and hydrogen peroxide. During the present year no sample was found to contain salicylic acid, and in only a few cases had hydrogen peroxide been used. The presence of benzoic acid and fluorides was not detected in any of the butters—boric acid being the only preservative present. All the samples of cream and butter from Norway, Sweden, Russia, and Denmark, were free from preservatives. None of the samples of margarine examined contained more butter-fat than is legally permissible, and an excessive quantity of water was found in one case only.

One hundred and forty samples of sheep-dips were received for examination: 20 were deficient in active ingredients, and in 63 other cases the formula required modification.

Linseed sometimes contains small quantities of glucoside, which is capable, under certain conditions, of producing hydrocyanic acid, although it is stated that the hot-press process of preparing linseed cakes destroys the enzyme. Results of an investigation showed that, when allowance is made for the oil removed during the pressing, the cake yields the same amount of hydrocyanic acid as the original seed.

Samples of oysters obtained from the West of England were examined in consequence of a suspicion that copper-poisoning had resulted from eating them. Many of the oysters had a distinct green appearance, and all contained copper and zinc, the largest amount of copper found being 1·22 grains of metallic copper per dozen oysters, and of zinc 2·31 grains. The oysters containing most copper were so distinctly green in colour that no person would have been likely to eat them. The growth of the oysters did not appear to have been affected by the presence of the two metals.

Ninety-three samples referred by magistrates under the Food and Drugs Acts were examined during the year. In 14 cases, including 9 samples of milk, the reports of the public analyst could not be confirmed. Certificates could not be issued with regard to 3 other samples of milk, owing to the high state of decomposition in the case of 2 of the samples, and because the bottle containing the other sample was burst in transit. The following are the particulars of some of the more important cases: *Butter.*—In one case where the charge was excess water, less than the legal amount was found. In 2 out of 3 samples alleged to contain foreign fat, the results obtained confirmed those of the public analyst; in the third case the butter gave results of an abnormal character, but having regard to the fact that similar results were obtained from the analysis of genuine butter from the same locality, it was impossible to say that the fat was not wholly butter-fat. One sample of butter, alleged to contain cocoanut oil and an excess of water, was found to contain excess of water, but no foreign fat. *Milk.*—Except in the case of 9 samples, the results obtained were in agreement wholly or partially with those of the public analyst; the charge in three of these cases was added water and in the other six abstraction of fat. *Malt Vinegar.*—One sample contained a large proportion of vinegar not derived from malt or grain, but in the case of the other sample received the results were not inconsistent with those given by genuine malt vinegar.

Whisky.—In 1 of the 2 samples received the alcoholic strength was found to be 21 degrees under proof, and was not therefore below the legal strength. *Margarine.*—The one sample received contained more than 10 per cent. of butter-fat. *Rice.*—The report of the public analyst, that the sample had been "faced," was confirmed. *Cream of Tartar.*—A sample condemned by the public analyst was found to comply substantially with the requirements of the British Pharmacopœia. The other samples referred consisted of rum, cream, and coffee, and in all these cases the results of the public analyst were confirmed.

The total number of samples submitted by the Board of Agriculture under the Fertilisers and Feeding Stuffs Acts was 47, consisting of 15 fertilisers and 32 feeding stuffs. The particulars relating to some of these samples were as follows: *Plant-Food.*—An article, sold in penny packets, and guaranteed to contain 22 per cent. of phosphates and 0·85 per cent. of potash, consisted mainly of fine earth and ashes with only 1·84 per cent. of phosphate and 0·33 per cent. of potash. *Tomato Manure.*—Contained only 4·7 per cent. of potash available for plant-food, although stated to contain "4·6 per cent. of free potash and 12·4 per cent. of combined potash." *Fish Manure.*—The sample examined consisted of fine ashes mixed with a few fish-bones, and contained only 0·59 per cent. of nitrogen and 2·4 per cent. of phosphates. The other fertilisers examined included superphosphates, wool-waste, bone-meal, and guanos, deficient in phosphates, nitrogen, or potash. *Cottonseed Meal.*—A sample described as "Double Decorticated Cottonseed Meal" consisted of the ground meal of undecorticated cottonseed cake, containing the whole of the cortex proper to the seed. *Milling Products.*—A sample of barley meal was found to contain 5 per cent. of sand. As much as 50 per cent. of ground-oat husks was present in a sample of "sharps." Other samples contained various quantities of tapioca meal and rice husks.

Details regarding the numbers of various articles examined are given in the Appendices to the Report. W. P. S.

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STUDY COMMITTEE OF THE INTERNATIONAL DAIRY FEDERATION.*

Lange Voorhout 88, the Hague,
October, 1912.

Dear Sir,—At the final general meeting of the Fifth International Dairy Congress, held at Stockholm, July 1, 1911, the following resolutions were carried:

"The next International Dairy Congress will fix the percentage of butter-fat in the dry matter which may be considered as normal figures for all sorts of cheeses which are found in the markets of the world."

"To secure the exactness of the result of the discussions, the Study Committee of the International Dairy Federation shall do the preparative work necessary specially to arrive at an international agreement on the methods for the analysis of cheese."

At the meeting of the Study Committee, held at Brussels on March 13 and 14, 1912, I was invited to prepare reports for this purpose, and I now beg to submit for your consideration proposals for the unification of the methods for the analysis of cheese.

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* Readers of the journal interested in this matter are invited to communicate with Dr. Swaving.

These proposals are the consequence of discussions of a special committee composed of delegates of cheese control-stations, of the Government dairy-station at Leiden, of municipal laboratories, of the Government agricultural experimental stations, and of myself as chairman. This committee has agreed to the methods for the analyses of cheese to be followed in the Netherlands.

I should feel obliged if you would kindly give these proposals your careful consideration, and let me know what modifications, if any, you wish to suggest.—

Yours faithfully,

SWAVING,

Secretary of the Study Committee of the International Dairy Federation.

PROPOSALS FOR THE UNIFICATION OF ANALYTICAL METHODS FOR CHEESE.

1. *Determination of Water in Cheese.*—Five grms. of cheese are weighed in a wide dish containing clean sand, and mixed and well ground up by means of a glass bar with flattened end, which has been weighed with the dish. The sample is then dried at the ordinary temperature—if possible *in vacuo*—for at least sixteen hours, and immediately afterwards dried for about one hour in an oven at 105° C., and weighed. Drying is continued, and weighing repeated with short intervals (half an hour).

2. *Determination of Fat in Cheese*—(*a*) *By Gerber's Method, with Modified Cheese Butyrometer* (*Van Gulik*) *for Three Grms. of Cheese.*—Three grms. of cheese are introduced with the aid of a funnel into the widened part of the cheese butyrometer, which has been previously half filled with sulphuric acid (sp. gr. 1·50). The weighing of very soft cheese is made in a small beaker. The butyrometer is then warmed in a water-bath at 65° to 70° C., and the casein dissolved, while shaking and swinging the butyrometer at intervals. When no more small particles of cheese are visible, the butyrometer is left in the water-bath for a short time, while swinging at intervals, and, after 1 c.c. amyl-alcohol has been added, it is filled with sulphuric acid up nearly to the division mark 35. The contents are mixed with care, not swinging the butyrometer oftener than necessary for a complete mixing, then left for about five minutes in

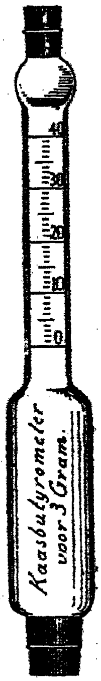

FIG. 1 (½ size).—GERBER-VAN GULIK BUTYROMETER.

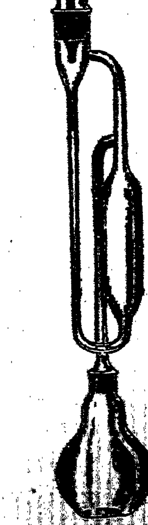

FIG. 2 (½ size).—SMETHAM FLASK.

the water-bath, and centrifuged for four minutes at a speed of 1,000 revolutions per minute. The diameter of the disc-plate of the Gerber centrifugal machine must be at least 47 cms. (the machine should be warmed when the temperature of the room is low). Finally, the butyrometer is placed for five minutes in a water-bath at 65° C., and the height of the fatty layer read at 65° C.

(*b*) *By the Hydrochloric Acid Method.**—One to 5 grms. of rasped cheese are gently boiled with 20 c.c. hydrochloric acid (sp. gr. 1·13) in a small flask, and shaking until all small particles have disappeared; then either Smetham's perforation method or Schmidt-Bondzynski's modified method is applied.

Smetham's Perforation Method.—The hydrochloric acid solution, when cool, is carefully introduced into the flask of Smetham's extractor, as modified for the estimation of fat in milk (Marius, Utrecht), and the flask filled with ether until it flows over. After two hours the ether is evaporated, and the fat dried for half an hour in the oven at 102° C. Drying is continued to constant weight. For control, a second flask can be connected to the extractor and treated as above.

Modified Schmidt-Bondzynski's Method.—The hydrochloric acid solution is carefully introduced into a mixing cylinder of 100 c.c. capacity, and, when cool, ether is added till the total volume is about 50 c.c. The cylinder is closed with a damped cork, then well shaken, and left for at least two hours to separate. The ether-fat solution is then drawn off as completely as possible (to at least 1 c.c.) in a weighed flask. A fresh quantity of 50 c.c. ether is introduced into the cylinder, and the contents swung several times. After one hour's rest the ether is again drawn off as completely as possible and added to the first ether-fat solution in the weighed flask. The ether is then distilled off, and the fat dried for half an hour in the oven at 102° C. Drying is continued to constant weight.

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REVIEWS.

THEORIE UND PRAXIS DER MASSANALYSE. Von ALEXANDER CLASSEN. Unter Mitwirkung von H. Cloeren. Leipzig. 1912. Akademische Verlagsgesellschaft, m.b.H. Pp. i-ix + 1-772. Price 32 marks.

The author of this work, it will be remembered, edited and revised two editions of Mohr's classical treatise "Titrimethoden." Recognising, however, that modern developments in chemical science have to a great extent modified our conceptions of the way in which chemical reactions occur, he decided that, instead of revising the seventh edition of Mohr's treatise, he would write an entirely new book, remodelled in accordance with modern theories. Professor Classen's reputation is at once a guarantee that the task which he set himself has been executed in a satisfactory manner; and of the work under review it may be said that, as a student's handbook, for which it is primarily intended, it is in most respects excellent, whilst it will also be found of use to the practising chemist.

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* In applying this method it is advisable to use within these limits a smaller quantity of fat cheese and a larger one of skimmed cheese.

Volumetric methods are, with very few exceptions, based on scientific principles so that they only became possible after stoichiometrical relations had been established. The pioneer in the elaboration of these methods was undoubtedly Gay Lussac. He it was who first devised methods of alkalimetry and acidimetry, as well as those of estimating chlorine, whilst the degree of accuracy of which these methods are capable first became evident after the classical work of Stas on the determination of the atomic weight of silver. A treatise such as the one under review should therefore be something more than an empirical guide book, and it will be conceded that Professor Classen has dealt liberally with the theoretical side of the subject. Indeed, in some cases the writer ventures to think that the text might have been shortened considerably with advantage to the reader. This applies particularly in the sections in which the methods are described, where many well-known and even self-obvious equations are given, including ordinary double decompositions. The sections dealing with the history and principles of volumetric methods and on the theory of indicators, although very lengthy, deserve special mention for their clearness and lucidity. Under indicators, however, something should, in the writer's opinion, have been mentioned of the tautomerism which some of these substances exhibit (*cf.* J. T. Hewitt, ANALYST, 1908, **33**, 85). The author is, however, throughout immersed in the ionic hypothesis, to the exclusion of any other.

The inorganic methods described form a useful collection, and many of them have been devised or checked in the author's laboratory at the Technical High School, Aachen, but it is surprising to find so few references to British work. There is an important point to which attention may be called—namely, that in no instance is the limit of accuracy of a method stated; nor are any of the methods of estimating one and the same substance compared as to their relative accuracy.

As regards empirical methods, the book is decidedly disappointing. Thus, twelve pages are devoted to a description of various modifications of the soap test for estimating the hardness of water, and about two and a half pages to alkalimetric and acidimetric methods for this purpose. After all this the author very properly remarks that the only reliable method of estimating hardness in a water is by a gravimetric analysis of the mineral matter. The estimation of reducing sugars by Fehling's solution is carried no further than the work of Soxhlet in 1880; and, indeed, the author seems unaware that anything has been done in volumetric analysis since that time, for he gives a reference to Wein's gravimetric tables, making no mention of the fact, however, that one of them—that referring to maltose—is inaccurate.

ARTHUR R. LING.

BLEACHING AND DYEING OF VEGETABLE FIBROUS MATERIALS. By JULIUS HÜBNER. London: Constable and Co., Ltd. 1912. Pp. xiii+434, including Index. Parts 18, with 96 illustrations. Price 14s. net.

It is necessary on occasion for the analyst to have some knowledge of the practical methods of procedure involved in certain industries. The work in question is one which will supply the necessary information in the case of cotton, so far as this can be imparted in such a manner. It opens with a section dealing with the vegetable fibres, including those manufactured artificially from cellulose. The following section deals with the analysis of water and its purification, and the analyst need not expect to gain special information from this or from the following one dealing with chemicals and mordants. The chapter on bleaching is, as might be expected, quite up to date, and gives in outline a satisfactory account of modern practice.

The important operation of mercerising is next considered, and it is pointed out that Persoz, in 1846, preceded Mercer in a description of the contraction, or shrinkage, of a cotton fabric under the action of caustic soda solution. It was not until 1895 that Thomas and Provost obtained a permanent lustre on cotton by mercerising under tension, although Lowe in this country had previously patented a similar process without apparently realising its importance.

A short chapter deals with the production of mineral colours on vegetable fibres, including, of course, the well-known khaki. The natural (vegetable) colouring matters are then considered, and also the artificial or synthetic colouring matters which have played such a leading part in modern dyeing. This section includes many practical details in actual manipulation, and a full account of those dyes which are applied to mordanting fibres and the so-called "oxidation" colours.

An account of the actual machinery used in dyeing operations then follows, and, finally, the testing of dyes and fibres is dealt with. This section, which is probably the one to which the general analyst will turn, occupies only thirteen pages, and therefore cannot deal in any detail with the subject under review, the actual tables concerned with the identification of dyestuffs only taking up about four pages in all. The question of the identification of fibres is also compressed into about one page. The section dealing with actual analysis can therefore only present in outline the methods which might be adopted in practice.

The work is primarily intended, as Professor Meldola points out in an interesting preface, as a practical textbook; and, so far as it is possible to compress detail into the space available, serves its purpose.

The illustrations are above the average, and indicate in a satisfactory manner the nature of the plant used.

W. P. Dreaper.

DECEMBER, 1912. Vol. XXXVII., No. 441.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE DETECTION AND ESTIMATION OF ARACHIS OIL.

By NORMAN EVERS, B.Sc., A.I.C.

(*Read at the Meeting, November* 6, 1912.)

DISCUSSION.

THE PRESIDENT said that this process would be found to be a great advance upon Renard's process in point of rapidity. In order, however, to ensure accuracy, it was essential that all the modifications mentioned by Mr. Evers should be adhered to.

Mr. R. Ross asked if it would be of any use to ascertain the refractive index of the residues obtained after cooling. He (Mr. Ross) had found this to be useful to a certain extent with butter containing coconut oil, though in that case its utility was limited owing to the variations in composition to which butter was liable. In the case of olive oil, which appeared to be more constant in composition, the refractive index might be more helpful.

Mr. P. A. E. RICHARDS asked how this process compared with Renard's, more particularly with regard to the time required for a determination.

Mr. L. M. NASH asked for a definition of "saponified" olive oil.

Mr. RICHMOND asked whether the factors for calculating the quantity of arachis oil were arrived at from the samples of arachis oil referred to in the paper or from a more extended series of observations. It seemed to him that in order to obtain accurate results the melting-points would have to be very carefully determined, and fully corrected, because, while the variation of the factor from 17 to 22 was appreciable, the difference of 2 degrees between the corresponding melting-points was only just outside the limits of experimental error.

Mr. W. B. SAVILLE said that he had used Bellier's process, and had found it to work very well, though when the proportion of arachis oil was considerable the results were liable to be low. He had usually found the melting-point of the fatty acids to be 73° C. He had tested almond, apricot, cotton-seed, maize, peach-kernel, castor, and sesame oils by Bellier's qualitative method, and in each case found a perfectly clear solution on standing for one to two hours at 17° to 19° C. Rape-seed oil invariably gave a turbidity due no doubt to the small percentage of arachidic acid present in the oil. Arachis oil could be determined with reasonable accuracy by the author's process when in admixture with these oils.

Mr. EVERS, in reply, said that he had not determined the refractive index of the crystals, but should think it quite probable that it would afford valuable information. The time required for a determination of arachis oil by this process was merely a few hours, and little attention was required, whereas the Renard process took considerably longer. "Saponified" olive oil consisted of the oil extracted from the marc after the larger proportion of the oil had been expressed. It contained a good deal of free fatty acid, and in order to remove this it was washed with alkali, which operation gave rise to the term "saponified." The factors were arrived at from the results yielded by the samples of arachis oil referred to in the paper. The melting-points had not been corrected for barometric pressure or the height of the column of mercury above the liquid, and this possibly might account for the difference between his figures and Mr. Saville's. In Bellier's process he had never found the melting-point (uncorrected) to be higher than 71° C. In his experience the melting-point could be determined accurately within half a degree. There was a good deal of difference in the proportion of fatty acids obtained when the melting-point was 71° C. and when it was 72° C., and this accounted for the difference in the factor.

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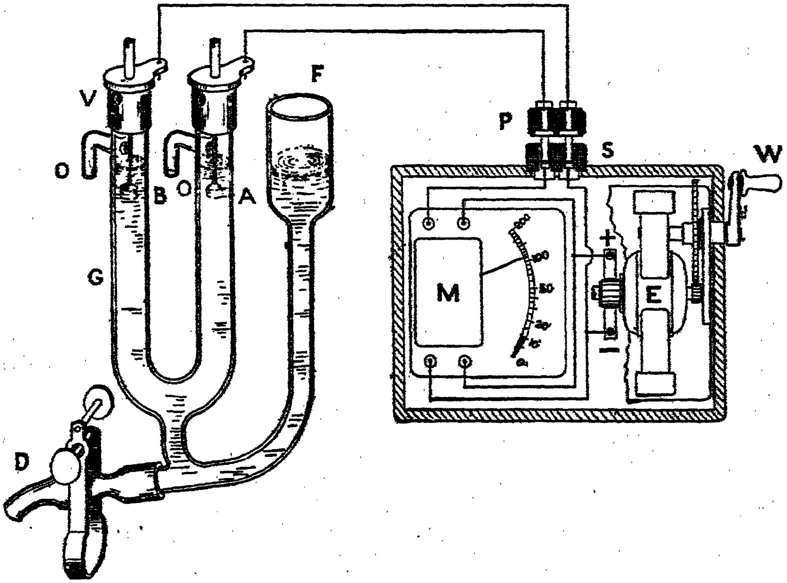
AN APPARATUS FOR TESTING WATER BY MEASUREMENT OF ITS ELECTRICAL CONDUCTIVITY.

BY L. ARCHBUTT, F.I.C.

(*Read at the Meeting, November* 6, 1912.)

AN apparatus known as the Digby and Biggs "Dionic" Water-Tester, designed primarily for detecting leakage in surface condensers, was recently brought to my notice; and having proved of some interest, I thought it might also interest some members of the Society. The usefulness of the apparatus depends upon the well-known fact that the conductivity of pure water is extremely small, and that the presence of any salt, acid, or base, in solution at once increases the conductivity. In very dilute solutions Kohlrausch found that the conductivity due to a given substance increases in proportion to the amount dissolved. For stronger solutions, however, this is not correct. Different salts also have different conductivities. The value of the apparatus depends upon its extreme sensitiveness in detecting mere traces of dissolved electrolytes, and in the rapidity with which a test can be made.

Referring to the diagram, the water to be tested is poured into the funnel *F*, and rises into the two arms of the U tube to the level fixed by the overflows *OO*. The electrodes *A* and *B* are short tubes of platinum-foil, and the current is supplied by the hand-driven dynamo *E*, fitted with a constant-speed friction clutch, which maintains a constant E.M.F. of 100 volts, within ¼ volt, when the handle *W* is turned at a rate faster than that at which the clutch is designed to slip. The current passing through the water is measured by the direct-reading conductivity meter *M*. Temperature is measured by a thermometer immersed in the water in the funnel. To make a test, all one has to do is to empty the U tube and funnel by opening the clip *D*, flush the apparatus with the water to be tested, then close *D* and continue pouring water into the funnel until it overflows at *OO*. Then turn the handle of the dynamo until the clutch is slipping, and at once read off the maximum deflection indicated by the needle.



The current generated is very small, not exceeding 0·004 ampère under normal conditions. The back E.M.F. due to polarisation of the electrodes is allowed for in the calibration of the meter, the scale units of which, ranging from zero to 2,000, represent approximately in value the reciprocals of megohms. The standard temperature for testing is 20° C., and a scale of corrections supplied with the instrument saves the trouble of adjusting the temperature of the water so long as it is between 10° and 40° C. It is therefore necessary only to take the temperature of the water and correct the meter readings by the corresponding factor.

The usefulness of the apparatus in a laboratory or works, and also its limits, will be apparent by the results of the following tests made in my laboratory:—

Distilled Water.	Conductivity (corrected to 20° C.).
Unboiled distilled water	2·7
Another sample	6·0
Another sample, after boiling to expel dissolved gases and cooling in air	1·5
Several samples thoroughly boiled in a silvered flask and cooled *in vacuo*	1·0 to 1·5
Unboiled water after passing CO_2-free air through for about one hour	1·0
Saturated with CO_2, and excess of gas shaken out at 20° C.	44·0

The above figures show that the ordinary distilled water of the laboratory, if boiled to expel CO_2, has a conductivity in this apparatus of 1·0 to 1·5. Derby Town water, with a soap hardness of 18·4°, total solids about 25 grains per gallon, chlorine about 1·15 grains, and SO_3 about 4 to 5 grains per gallon, had a conductivity of 552. 3 c.c. of this hard water diluted to 500 c.c. with boiled distilled water, and containing about 0·15 grain of total solids per gallon, had a conductivity of 4·2. This water gave a faint opalescence with silver nitrate in the cold, which disappeared on heating, and no perceptible precipitate with barium chloride, from which it is evident that a slight leakage of the hard water into the distilled water through a crack in the tin condensing worm, as sometimes occurs, would be detected by the conductivity meter rather more readily than by the silver nitrate test, and the test would be quantitative.

In the next table I give the conductivity of a few public water supplies.

	Conductivity (20° C.).
Glasgow, from Loch Katrine	34
Manchester, from Thirlmere	48
Blackburn	65
Birmingham, from Rhayader	80
Dewsbury	109
East Surrey water, from wells in the chalk, softened by Clark's process	135
West Middlesex water (Metropolitan Water Board)	390
Derby: mixture of spring water and Derwent Valley water (hardness, 11·9 degrees)	380
Derby: spring water only (hardness, 18·4 degrees)	552

The above, except the Derby samples, are from a pamphlet published by Messrs. Evershed and Vignoles, Ltd., the makers of the apparatus.

As there is a tendency for an apparatus of this kind to be offered as a substitute for chemical analysis, I have prepared a few solutions and tested their conductivity.

The saline solutions (except the $MgSO_4$) were made with the salts usually met with in concentrated boiler waters. Being made of equal strength, they show how the conductivity varies with the different salts, also that with solutions of the strengths stated the conductivity is not proportional to the amount dissolved (compare Na_2SO_4).

The acids and bases show enormous variations; the first three, also the caustic soda solution, were made up so as to have about the same conductivity as a solution of common salt containing 50 grains per gallon; the acetic acid and ammonia were random mixtures. I have stated all the strengths in "normal" terms as well as in grains per gallon for convenience of comparison. The figures show how greatly the conductivity of the water tested may be influenced by a slight acidity or alkalinity.

Some Saline Solutions.	Grains of Anhydrous Salt per Gallon.	Conductivity (20° C.).
$NaCl$	50·0	1,287
Na_2CO_3	50·0	1,236
Na_2SO_4	50·0	1,020
Na_2SO_4	100·0	1,751
$NaNO_3$	50·0	900
$MgSO_4$	50·0	954
Acids.		
HCl } 1·6 c.c. normal acid diluted to 500 c.c.	8·16	1,260
H_2SO_4 } 1·6 c.c. normal acid diluted to 500 c.c.	10·98	1,127
HNO_3 } 1·6 c.c. normal acid diluted to 500 c.c.	14·37	1,255
CH_3COOH, 26·5 c.c. N. acid diluted to 500 c.c.	222·60	400
Bases.		
$NaOH$, 3·2 c.c. N. soda diluted to 500 c.c.	17·9	1,214
NH_3, 35·45 c.c. N. ammonia diluted to 500 c.c.	84·7 (174·5 NH_4OH)	325

Lastly, non-electrolytes, such as cane-sugar, might be present in water in large quantity, and would entirely escape detection. Within limits, however, and applied to specific purposes, the apparatus is extremely useful, as is shown by the following tests:

CONCENTRATED WATER FROM STIRLING BOILER.

Total Solids, 130° C.	Non-Volatile Solids.	Chlorine as Chloride.	Conductivity (20° C.).
Grains per Gallon.	Grains per Gallon.	Grains per Gallon.	
19·8	19·0	3·6	445
51·9	47·9	14·3	1,113
53·7	52·1	16·7	1,166
60·1	54·2	17·5	1,261
69·1	64·0	11·7	1,272
73·3	67·2	16·0	1,431
75·6	72·2	18·8	1,579
112·4	108·8	24·2	[illegible]

The above samples were taken on various dates, and do not represent any particular series. They are arranged in order of conductivity. The water used in the boiler was partly condensed water and partly a hard water used as "make-up." The conductivity, though not strictly proportional to the non-volatile solids, is fairly proportional, whilst the chlorine is not. Suppose it were desired, for example, to keep the concentration of the water in this particular boiler below 100 grains per gallon in order to prevent "priming," it is evident from the above figures that the conductivity test is a better, simpler, and more rapid indicator of the amount of saline matter in solution than is the chlorine, which is about the simplest analytical test that could be made.

It is obvious that this apparatus may be extremely useful for detecting leakage of surface condensers. Condensed water from the hot wells of condensing engines will have a quite low conductivity, if there is no leakage, say 3 to 5. Cooling water will generally be hard, and will have a much higher conductivity. This applies especially to sea-water, the conductivity of which is about 50,000 on the scale of this instrument. Having ascertained, therefore, the conductivity of the cooling water used, and of mixtures of this with distilled water, a curve can be constructed from which the percentage of leakage water corresponding to any observed conductivity can be read off.

DISCUSSION.

Mr. F. EVERSHED asked whether the President had found this apparatus to be useful for controlling the addition of chemicals in softening water—one of the purposes for which the makers claimed that it might be used with advantage in place of the soap test. He (Mr. Evershed) should think that in simple cases, where the addition of lime only was required, the apparatus could be so used; but in more complicated cases, when soda, with or without lime, was used, the conductivity would not necessarily be diminished in the final result, though even here such a method of testing might be found useful, if applied with discrimination. In sugar-analysis a conductivity test would often measure inorganic impurities with sufficient accuracy and far more quickly than an ash determination. This last idea was not new, a paper on the subject having been read by Mr. Hugh Main at the International Congress of Applied Chemistry in 1909, but Mr. Main used the Kohlrausch apparatus, which required a supply of electricity from an outside source, while the apparatus now described had the advantage of being self-contained, the current being produced within the box by merely turning a handle. Various other applications of this instrument would no doubt suggest themselves.

Mr. RAYMOND ROSS said that in Lancashire there were many streams which, in addition to their ordinary main flow, received colliery pumpings, and also varying amounts of peaty water from the surrounding hills. Such water often had to be used for boiler purposes, and the variations in its composition presented a problem in regard to which the apparatus referred to might perhaps be useful.

Mr. G. N. HUNTLY said that the principle on which this apparatus was based had already been applied in practice for some time. There is a well-known oil-removing plant, in which the removal of oil from condensed water is effected by passing a continuous current between iron plates, the oil being carried down by the oxide of iron that was thus formed. It is found, incidentally, that if a fuse is placed in the circuit, any leakage of cooling water into the condenser is infallibly indicated by the blowing out of the fuse. The difference in conductivity was so great that a heavy fuse is readily blown out.

Mr. W. T. Burgess said that the conductivity measurements were, of course, mainly influenced by the mineral constituents, and gave no indication of the organic purity, or otherwise, of the samples tested.

Mr. J. H. B. Jenkins said that the apparatus used by Mr. Main answered very well for the estimation of ash in sugar, the determinations being made very quickly. It was quite easy to experiment in this direction without any special apparatus beyond that which was ordinarily at hand in the laboratory. If the terminals of an induction-coil were connected with a Wheatstone bridge—the galvanometer being replaced by a telephone receiver—and if into the resistance space of the bridge were introduced a platinum dish with a small electrode kept about ⅛ inch away from the bottom of the dish, samples of water could be introduced into the dish one after the other and tested by noting when the sound in the telephone receiver disappeared. In this case, of course, it was the resistance and not the conductivity of the water that was measured.

The President, in reply, said that he could quite understand that there might be circumstances in which this apparatus would be useful in connection with water-softening, but, on the other hand, conditions could easily be imagined in which it would be practically useless. Probably in a case such as that mentioned by Mr. Ross, if the conductivities of the different sources of water were known, it would be possible to determine approximately the extent to which the various kinds of water were present, but here again there would be limitations.

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THE EXAMINATION OF CHINESE AND JAPANESE WOOD OIL.

By A. CHASTON CHAPMAN, F.I.C.

(*Read at the Meeting, November* 6, 1912.)

Chinese Wood Oil.

During recent years the trade in Chinese wood oil has assumed considerable proportions, and its chemical examination has consequently become a matter of importance both to buyers and sellers. This fact, coupled with the very interesting nature of the oil itself, has induced a number of chemists to devote themselves to its investigation, and a number of papers dealing with the general chemistry and analytical examination of the oil have been published in English, American, and Continental journals. Notwithstanding this, it cannot be said that chemists are in complete agreement as to the properties which should characterise the genuine oil, and for some time past trade disputes culminating in arbitration cases have been of somewhat frequent occurrence. Having for several years been much interested in the examination of this oil, I have thought that it might not be without interest if I were to place some of my results and conclusions before this Society.

This oil is obtained, as is well known, from the seeds of the *Aleurites cordata*—a tree which grows extensively in China and other parts of the East. Other species of Aleurites—as, for example, *Aleurites Fordii*—also occur, and it is probable that the commercial oil often represents a product obtained from more than one species. I shall revert to this later.

According to a recently-published United States Consular Report, it would appear that Szechuan oil is preferred in Hankow on account of its lighter colour, and that the province of Szechuan accounts for 30 per cent. of the total production, Hunan and Kweichow producing 50 per cent., and Hupei (chiefly in the region of Ichang) 20 per cent. Lewkowitsch ("Chemical Technology and Analysis of Oils, Fats, and Waxes," fourth edition, vol. ii., p. 60) states that, in the preparation of the oil, the seeds are roasted in a flat dish over a naked fire, are then crushed between stones, and the oil expressed from the crushed mass in primitive wooden presses. This account of the preparation of the oil is confirmed by several independent statements made to me by persons who have resided for a considerable time in China, and who are familiar with the method of expression adopted by the natives. This crude method of obtaining the oil, considered in conjunction with the fact that the production is in the hands of a large number of very small producers, and that the oil in question is a powerful drying oil, is of considerable importance, since it affords a partial explanation of the somewhat widely varying analytical results which have been obtained by chemists in the examination of this oil, and which have been recorded in the literature of the subject. The small quantities of oil produced by the various individuals are collected by Chinese middlemen, and sold to merchants in Hankow, who clarify the oil in tanks, and then pack it for shipment. In colour the oil varies very considerably, the dark oils having as a very general rule a more markedly unpleasant taste and smell than the paler ones.

The analytical determinations which are of the greatest importance are the specific gravity, the iodine value, the refractive index, the viscosity, and the manner in which the oil polymerises when heated. I propose to refer in the first place to these estimations, and to deal later in the paper with several supplementary methods of examination.

Specific Gravity.—Since the specific gravity of Chinese wood oil is higher than that of any other fatty oil, with the exception of castor oil and possibly of tallow-seed oil, the determination of that number is clearly of great importance. The numbers recorded in literature vary somewhat considerably, but many of the earlier ones are of less value than they might otherwise have been owing to some apparent confusion between the Chinese and the Japanese oil, and also to the fact that in some cases the nature of the ratio oil : water is not clearly stated. Among later and more reliable numbers may be mentioned those of Lewkowitsch ("Chemical Technology and Analysis of Oils, Fats, and Waxes," fourth edition, vol. ii., p. 63) 0·9412 to 0·9418; Kreikenbaum (*J. Ind. and Eng. Chem.*, 1910, **2**, 205), 0·9401 to

0·9454; and F. Browne (*Chem. News*, 1912, **106**, 15), 0·9402 to 0·9431. Jenkins (ANALYST, 1898, **23**, 113), in a paper communicated to this Society, gives 0·9343 and 0·9385 for two samples; but as he uses the terms "Japanese wood oil," "Chinese wood oil," and "tung oil," as synonymous, the value of these numbers is considerably diminished. Rowland Williams (*J. Soc. Chem. Ind.*, 1898, **17**, 304), in a paper entitled "Japanese Wood Oil," gives numbers ranging from 0·9413 to 0·9432, and it is tolerably clear from these and the other results he gives that these were samples of the Chinese oil. Boughton (*Seventh Int. Cong. App. Chem.*, 1909, Section **1**, 89) records numbers for five samples of China wood oil which he had received directly from the United States Consul at Hankow, and for two further samples which had been pressed for him from the nuts. Six of these gave numbers ranging from 0·9408 to 0·9428 at 15·6° C., but one of the five samples from Hankow had at the same temperature a specific gravity of 0·9346, which is far below any number recorded by other observers for the Chinese oil, and which must tend to throw considerable doubt on the purity of the specimen.

Iodine Value.—Since this number depends so largely on the precise method adopted and upon the conditions under which the test is carried out, and as this information is not given in many of the earlier papers, it would serve no useful purpose to refer in detail to the numbers recorded, but it may be said that numbers varying from 150 to 170 have been given. In some cases, however, it is probable that the Japanese oil had been used. Boughton (*loc. cit.*) has made a special study of the extent to which the iodine value varies with different experimental conditions, and finds that variations in temperature have much less effect on the Hubl than on the Hanŭs method. For several reasons I prefer the Wijs method, and the iodine values recorded in the table given below have been determined in the following manner:—About 0·1 grm. of the oil was taken, dissolved in 20 c.c. of purified carbon tetrachloride, 30 c.c. of the ordinary Wijs solution were added, and the reaction allowed to proceed for three hours in the dark.

Refractive Index.—Since the refractive index of wood oil is considerably higher than that of any other fatty oil, the determination of that value is of the greatest importance from the analytical point of view. Comparatively few numbers are recorded in the literature of the subject, but it may be mentioned that Boughton gives 1·5238 at 15·6° C., which is approximately equal to 1·521 at 20° C., the temperature at which my own observations have been made.

Viscosity (Time of Efflux).—As the viscosity of tung oil is considerably higher than that of any other fatty oil which would be likely to be used as an adulterant, a determination of that number is very useful, although, as will be seen from the results recorded below, there are wide variations even in the case of oils of undoubted genuineness. It must not be overlooked, moreover, that the viscosity of wood oil may be very considerably increased by heating it to a temperature short of that required for its solidification. The viscosities given in the following table have been determined in a standard Redwood viscosimeter at 15·5° C. From a circular recently issued by the New York Produce Exchange, it would appear that C. V. Bacon has made a large number of viscosity determinations, using a Tagliabue instrument, and that he attaches considerable importance to the results.

Heat Polymerisation Test.—One of the most interesting and characteristic properties of wood oil is that of solidifying to a jelly when heated to a sufficiently high temperature, a change which appears to be due to the polymerisation of the glyceride of elæomargaric acid, of which the oil largely consists. Since the hardness of the resulting jelly is considerably diminished by the addition of other fatty oils, it will be seen that a test based upon this property is capable of yielding very important indications. Several attempts have been made to make such a test quantitative. Thus, in a circular issued by the New York Produce Exchange, to which I have referred already, a method devised by C. V. Bacon is tentatively put forward as a method to be adopted in the United States. Since this test has not, so far as I am aware, been published in any scientific journal, it may be well to give the following description of it:

"Into a test-tube of ¾ inch diameter and 4 inches in length there are transferred about 10 c.c. of pure China wood oil; into another test-tube there is transferred a similar volume of pure China wood oil adulterated to the extent of 10 per cent. A sample of the oil to be tested is treated in a like manner, and these are placed in a proper support and immersed in an oil bath which has a temperature of about 288° C., so that when the tubes are in it a temperature of 280° or 285° C. (maximum) can be maintained. The oil bath containing the tubes is maintained at this temperature for exactly nine minutes, the tubes are then withdrawn and the test sample is compared with the pure oil, and the same oil adulterated with 5 and 10 per cent. of foreign oil. After the tubes are withdrawn from the oil bath, each tube should be stabbed from the top to the bottom with a small bright spatula. Pure oil will give a hard, clean cut, and when the knife is withdrawn the incision will look like a straight line, while an oil having as low as 5 per cent. adulteration will invariably be softer, and the incision will have a peculiar feathered effect; an adulteration of 10 per cent. will be soft and pushy, while an adulteration exceeding 12 per cent. in many instances will remain entirely liquid."

In the same circular a further test is described as being one at present used by New York importers and varnish-makers. This is described in the following words:

"For Hankow and Shanghai wood oil, 100 grms. should be heated in an open basin (6 inches in diameter) as soon as possible to a temperature between 540° and 560° F., and if it solidifies in about six to six and a half minutes, cuts dry, and is firm in body, without discoloration and without being sticky, it should be passed as a good delivery. For Canton and Hong-Kong wood oil deliveries, the time should be from four and a half to five and a half minutes in an open basin as above. Should a longer time be taken by presumably pure wood oil, other tests confirming purity shall be positive."

This latter test is, in my opinion, an unsatisfactory one, since it is impossible to control the high temperatures necessary or to keep them at all constant, owing to the shallowness of the containing vessel and the large cooling surface. As soon, moreover, as solidification commences, it is impossible to continue the stirring, with the result that, if the application of heat be continued, the bottom portions of the oil will char, whilst the top portions are still in a soft and sticky condition. The main objection to Bacon's method lies in the fact that it involves comparison with a standard sample, which in turn implies that wood oil is practically constant in its polymerising properties. If all genuine wood oils were known to polymerise in precisely the same manner, there would be some justification for drawing definite conclusions from the indications furnished by such a method, although even then they could not, from their very nature, be more than roughly quantitative. There can be no doubt, however, that such is not the case, and consequently it is not wise to attempt to draw too definite conclusions from the results of the heat test, valuable as its indications are. It may be mentioned in this connection that F. Browne (*loc. cit.*) has described a method of applying the heat test which is, in my opinion, superior to that of Bacon, and less open to criticism, inasmuch as he recognises that genuine oils may vary somewhat in their setting properties; but the temperature to which the oil is heated is so high that the differences between the setting period of a genuine oil and one adulterated with, say, 10 per cent. of some other fatty oil is only two or three minutes. As the result of a considerable amount of experience, I have been led to attach more importance to the hardness of the jelly obtained under standard conditions than to the time required for bringing about polymerisation, and I have devised the following method of carrying out the test, which is capable of yielding definite and concordant results:

About 5 c.c. of the oil to be examined are introduced into each of two test-tubes 6 inches long by $\frac{5}{8}$ inch diameter. These are then immersed in a bath containing melted paraffin wax at a temperature of approximately 100° C. The temperature of the bath is then raised to 250° C., taking about fifteen minutes for the operation. As soon as that temperature is reached the time is noted, and the source of heat adjusted so that the temperature of the bath is maintained constant at 250° C. At the end of half an hour one of the tubes is withdrawn, allowed to cool, and, when cold, is broken, and the jelly examined. The other tube is kept in the bath at 250° C. for a further period of half an hour, at the end of which time it also is withdrawn and allowed to cool; it is then broken, and the hardness of the jelly observed. Chinese wood oil of good quality should give at the end of half an hour a fairly firm jelly, which, at the end of one hour, should become quite hard. It is advisable in all cases to carry out comparison tests alongside of the oil under examination, using for the purpose a sample of oil known to be of good quality.

I have not found it possible to express the hardness of the solidified cylinders by means of numbers, but with a little experience it is very easy to distinguish between a sample of genuine oil and the same oil containing a small percentage of some fatty oil, such as soya bean or sesame. In referring to the polymerisation experiments, I have used the words "very hard," "hard," and "fairly hard," to denote the consistency of the polymerised oil, since such expressions are quite sufficient for the purpose. In addition to the degree of hardness of the solid cylinders of oil, some attention should be given to their physical characters. When cut with a knife or broken across, the cut or fractured surface should be smooth and free from stickiness, and small portions when rubbed in the hand should break down completely into a soft crumbly mass, which should not adhere to the fingers.

In the following table I give the results of the examination of seventeen samples of Chinese wood oil from Hankow:

Sample.	Iodine Value.	Sp. Gr. 15°/15° C.	Saponification Value.	Refractive Index at 20° C.	Time of Efflux at 15·5° C. Seconds.	Polymerisation One Hour at 250° C.
No. 1	169·9	0·9419	196·6	1·5207	2178	Very hard.
,, 2	168·4	0·9406	193·8	1·5181	1636	Hard.
,, 3	166·5	0·9426	194·3	1·5190	1946	Fairly hard.
,, 4	166·4	0·9417	193·0	1·5170	1880	,,
,, 5	168·8	0·9430	195·6	1·5195	2017	Very hard.
,, 6	170·0	0·9440	194·5	1·5180	1849	Hard.
,, 7	168·6	0·9416	193·0	1·5150	—	Fairly hard.
,, 8	171·0	0·9414	192·0	1·5170	—	Hard.
,, 9	169·7	0·9437	194·1	1·5176	1997	,,
,, 10	173·0	0·9420	192·5	1·5165	1722	,,
,, 11	176·2	0·9417	192·0	1·5168	1605	,,
,, 12	172·6	0·9429	196·0	1·5180	1740	,,
,, 13	174·2	0·9427	194·6	1·5182	1690	,,
,, 14	173·7	0·9430	195·0	1·5194	1820	,,
,, 15	172·8	0·9440	194·6	1·5193	2047	,,
,, 16	169·5	0·9420	195·2	1·5160	1804	,,
,, 17	169·6	0·9433	195·2	1·5187	1820	Very hard.
Average ...	170·6	0·9425	194·2	1·5179	1850	

On reference to the above table it will be seen that the specific gravities of the seventeen oils in question range from 0·9406 to 0·9440, and these results, which are confirmed by a very much larger number, may be regarded as approximately the limits of variation for the genuine oil. I am not prepared to say that no pure Chinese wood oil has a specific gravity below the lower limit mentioned above, but a result appreciably below 0·940 is in itself sufficient to cause a sample of oil to be regarded with suspicion.

With regard to the iodine value, it will be seen that the average of the results is 170·6, and it may be taken that a genuine oil should not give numbers differing from that by more than a few units, using the Wijs method, and adopting the conditions I have specified.

The saponification value is not, in the majority of cases, of very much importance; but as it may sometimes be useful, it should always be determined. It is interesting to note that my limits agree fairly closely with those recorded by Rowland Williams (190·7 to 196·1), in the paper to which reference has already been made.

The refractive index rarely falls below 1·517, and an oil giving a result below 1·515 at 20° C. should be regarded with suspicion.

As I have already pointed out, the viscosity of this oil appears to vary within somewhat wide limits, but the numbers I have given will suffice to indicate the kind of result which may be expected in the case of genuine oils.

A paper dealing with the oil derived from the seeds of *Aleurites Fordii* having appeared in the Bulletin of the Imperial Institute (1907, 5, 134), I wrote to Professor

W. R. Dunstan asking him if he could let me have some of this oil. Although he had none of the oil left, he kindly supplied me with a quantity of the nuts of *Aleurites Fordii*, as well as with some of the *Aleurites cordata*, and I desire to take this opportunity of expressing to him my thanks for this courtesy. From each of these I prepared a quantity of oil by petroleum extraction, and found that the former had a specific gravity of 0·9427, and the latter of 0·940. Both these oils, however, were abnormal in respect of their polymerising properties, and for that reason I have not included the other analytical results. Whether this abnormality is due to the method of extraction adopted or to the fact that the nuts were several years old, I am not prepared to say, but it is certain that both oils were markedly inferior in their polymerising power to any genuine sample of the commercial oil that I have examined. This result gives point to the remark I made in criticising Bacon's method, that all samples of genuine wood oil cannot be expected to polymerise in exactly the same manner. There is no doubt that Chinese wood oil, like all other oils, varies within certain limits, both in respect of its chemical and physical properties. As a matter of fact, botanical and geographical origin, climatic variations, differences in treatment during the manufacture of the oil, and the duration and conditions of storage, unquestionably affect its properties, and I am, therefore, very strongly of opinion that it is not advisable in determining its purity to adopt any single method which involves a comparison with an oil possessing certain properties in a maximum degree. Of course, if any purchaser desires to buy wood oil on the results of the polymerising test alone, I see no reason why he should not do so. It must not be overlooked, however, that it may quite well happen that oil which does not reach a certain high polymerisation standard may nevertheless be free from adulteration.

Supplementary Tests.

Drying Test.—About 0·3 to 0·4 grm. of the oil is spread in a thin film on a glass plate and heated in an air-bath for one hour at a temperature of 105° C. The genuine oil dries completely to a crinkly, rubber-like substance, which can be removed without difficulty from the glass surface, and which is free from softness or oiliness. Under these conditions the oil gains from 2 to 3 per cent. in weight.

Iodine Test.—As is well known, if a solution of iodine in chloroform or some other suitable solvent be added to the oil, almost immediate solidification takes place. This is a very useful qualitative test, and McIlhiney (*J. Ind. and Eng. Chem.*, 1912, 4, 497; ANALYST, 1912, 410) has recently been carrying out experiments with the object of ascertaining whether it might not be made quantitative. In the method he has put forward tentatively, an excess of a solution of iodine in glacial acetic acid is added to a weighed quantity of the oil dissolved in the same solvent. The products of the reaction are then extracted with petroleum ether, and the amount of oil which has not undergone solidification, and which is soluble in the petroleum, is weighed. I have made a few experiments with this process, and have obtained results which appear to indicate that when this test is fully worked out it will be found to be a very useful one.

Optical Activity.—A number of samples of oil which I have examined have been practically inactive, although in one case a slight dextro-rotation (+ 1° for a

100 mm. tube and sodium light) was observed. Tallow-seed oil, on the other hand, is known to be appreciably lævo-rotatory.

Bromine Thermal Value.—This number was determined by the method recommended by Hehner and Mitchell—viz., by adding 1 c.c. of bromine to a solution of 1 grm. of the oil in 10 c.c. of chloroform, the mixture being kept in a vacuum jacketed test-tube. The following results were obtained in the case of six samples of the oil:

	Rise in Temperature. °C.	Iodine Value. / Rise in Temperature.
Sample A	24·10	7·00
Sample B	24·76	6·80
Sample C	25·57	6·51
Sample D	24·98	6·92
Sample E	24·72	7·00
Sample F	24·56	7·17

For purposes of comparison I give below the values obtained in the case of three samples of the Japanese oil:

	Rise in Temperature. °C.	Iodine Value. / Rise in Temperature.
Sample A	24·70	6·40
Sample B	23·10	6·45
Sample C (prepared in laboratory)	24·50	6·27

It will be seen that the average value (6·9) obtained by dividing the iodine value by the rise in temperature is substantially the same as that given by Jenkins (*loc. cit.*) in his paper on wood oil. There appears, however, to be no reasonable doubt that the two samples of oil with which Jenkins worked were Japanese oil, and for this it will be seen that my average value is somewhat lower—viz., 6·37. In both cases, however, the value is appreciably higher than with the great majority of fatty oils.

Hexabromide Test.—It has been shown independently by Hehner and Mitchell, and by Jenkins, that when wood oil is treated with bromine under the conditions laid down by the former authors (ANALYST, 1898, **23**, 310), no ether-insoluble bromide is formed. I have confirmed the correctness of these observations, working with the seventeen samples of Chinese wood oil referred to above, and with four samples of the Japanese oil. In no case was there the slightest formation of precipitate, even when the flask containing the mixture was kept in iced water and allowed to stand for twenty-four hours. On the other hand, perilla, candle nut, and tallow-seed oils all yield more or less hexabromide; and as these oils are possessed of high drying properties, and are produced in large quantities in China and the East, the test may be one of considerable importance, and is, in any case, one which should always be applied. In addition to the above-mentioned oils, it would, of course, aid in the detection of linseed oil. Tea-seed oil, as might be expected, does not yield any insoluble bromide. I have been able to detect with considerable ease the presence of 10 per cent. of perilla oil, but it should be mentioned that this oil yields a much larger proportion of the hexabromide than any of the oils mentioned above, with the possible exception of linseed.

The oil should, of course, be tested in all cases for those fatty oils such as cottonseed and sesame, which give characteristic reactions. I may mention, in this connection, that although I have only in one instance detected cotton-seed oil in Chinese wood oil, I have obtained the Baudouin reaction for sesame oil in a considerable number of cases. Genuine China wood oil does not give this reaction, nor does tea-seed oil. In a few instances in which the reaction was very faint it is possible that it may have been due to the fact that the wood oil had been tanked in a vessel which had previously contained sesame; but in the other cases there can be no doubt that sesame oil had been used as an adulterant, particularly as the majority of these samples were met with at a time when wood oil was commanding an unusually high price.

JAPANESE WOOD OIL.

As I have already indicated, many observers have regarded this oil as being practically identical with Chinese wood oil, and have referred to the two indiscriminately under the common designation "tung oil." Whilst resembling Chinese wood oil somewhat closely in its general characters, it appears to be chiefly obtained from the nuts of a different tree—viz., the *Paulownia imperialis* (Japanese *Kirinoki*), although it is not improbable that other seeds may occasionally be employed. The chief producing centres appear to be Wakasa and Echizen. Very little of this oil is exported from Japan, almost the whole being required for home consumption; and its chief interest to the analyst lies in the fact that the two oils are not equally suitable for certain manufacturing purposes, and that accidental or intentional substitution of one for the other may occasionally occur. I give below the results of the examination of three samples, all of which were obtained through the courtesy of the British Consul-General in Kobe:

Sample.	Iodine Value.	Sp. Gr., 15°/15° C.	Saponification Value.	Refractive Index at 20° C.	Time of Efflux at 15·5° C. Seconds.	Polymerisation Two Hours at 250° C.
No. 1 ...	158·0	0·9377	195·2	1·5083	1230	Soft.
No. 2 ...	149·0	0·9400	193·4	1·5052	1620	Soft.
No. 3 ...	151·8	0·9349	196·3	1·5034	—	Very soft.

"No. 1" was produced in Wakasa, whilst "No. 2" came from Idzumo. As to the district from which "No. 3" was derived, I am not quite sure.

In consequence of a letter which I wrote to the British Consul at Kobe, I received through the kindness of Messrs. Jardine, Matheson, and Co., Ltd., a quantity of the nuts of the *Paulownia imperialis*, from which the Japanese oil is made. A quantity of the oil was prepared from these nuts by extraction with light petroleum, and gave on analysis the following results:

Specific gravity (15°/15° C.)	0·9351
Iodine value	153·5
Saponification value	193·5
Refractive index at 20° C.	1·5050
Bromine thermal value (rise in degrees Centigrade) ...	24·5

When heated for two hours at a temperature of 250° C., this sample had not solidified, but was still a viscous oil. On reference to the above numbers it will be seen that with the possible exception of "No. 2" the specific gravities are very appreciably below those of the Chinese oil, which confirms the statements of Lewkowitsch and other observers. The same applies to the iodine values and refractive indices. Even greater than these differences, however, are the differences in polymerising properties, the Japanese oil remaining quite liquid under conditions such as suffice to convert the Chinese oil into a very hard jelly, which crumbles when rubbed between the fingers and becomes a dry powder.

My best thanks are due to my assistant, Mr. Frederick T. Harry, for his valuable help in connection with this work.

DISCUSSION.

Mr. HEHNER remarked that when the fatty acids from this oil were dissolved in acetone and cooled, magnificent crystals resulted. This property was possessed by no other fluid oil. The crystals retained their form as long as the vessel was kept closed: when it was opened they generally collapsed immediately into a jelly-like mass. Another remarkable feature of wood oil was that it was not capable of forming a hexabromide like other oils having a high iodine absorption—for example, linseed oil and fish oils—which might be used as adulterants. Probably the differences in the extent to which polymerisation took place were due to small quantities of botanical impurities which exercised a catalytic action.

Mr. J. H. B. JENKINS said that when he examined these oils fifteen years ago the quantity of them on the market was very small, and consequently his investigation had been much more limited than that of Mr. Chapman. He should like to emphasise what Mr. Hehner had said as to the bromine addition value: the absence of any insoluble brominated compound markedly distinguished these oils from other drying oils. It was the indirect result of Mr. Hehner's and Mr. Mitchell's valuable work on the bromine reactions that had called his attention to the peculiarities of these oils. Following those authors' method, he had compared the iodine values and heat of bromination of a wide range of fatty oils. The usual factor for converting the one into the other was found, except in the case of a strange oil which was put into his hands by a friend as "Japanese Wood Oil." In that case, however, the factor was distinctly abnormal, and led him to think that iodine must have some exceptional action on the oil. He consequently tried the effect of adding a saturated solution of iodine to the oil dissolved in chloroform, and, whilst stirring, he found the whole was converted into a jelly. The other abnormal feature disclosed in his early examination of those oils was the very high refractive index. Both samples he had dealt with (ANALYST, 1898, 23, 113) were supplied as "Japanese Wood Oil"; but at that time, in common with many others, he had looked upon Japanese Wood Oil and Chinese Wood Oil as the same.

Mr. E. R. BOLTON asked what was the lowest iodine value that Mr. Chapman had found in what might be accepted as genuine wood oil. In making some experiments with a sample of Chinese wood oil which he believed to be genuine, but of which the iodine value was very low, he had found that with this oil the time required for complete absorption was very short, about a quarter of an hour's contact with Wijs' solution being sufficient, so that some of the low figures that had been published could hardly be attributed to insufficient time being allowed for absorption.

Mr. R. Ross remarked that the specific refraction $\frac{(\mu-1)}{D}$ of the various oils referred to in the first table worked out at 542 to 550, and of those in the last table at 537 to 538, the figure for the oil expressed by Mr. Chapman himself being 534. He had found the McKinley process of bromine absorption to be very useful, since it showed both the addition products and the substitution products, and very often enabled one to account satisfactorily for certain differences that were observed.

Mr. L. M. Nash said that at the present moment the iodine value by itself was of no assistance in judging wood oil, since linseed oil was about £4 per ton cheaper than wood oil. Mr. Chapman's figures confirmed his own views as to the refractive index, specific gravity and viscosity, all of which should be as high as possible. As to heating tests, he agreed with Mr. Chapman's criticism of the American method of heating to a very high temperature in a shallow dish, but he had found it useful to make a test by heating about 5 ounces of the oil in a dish of standard size at a temperature about 100° F. lower than in the American test, and noting the time that it took to reach the condition in which the oil was used in the factory.

The President asked whether Chinese and Japanese wood oils were ever mixed together. If this were done it would probably render the detection of adulterants more difficult.

Mr. Chapman, in reply, said that the difficulty of detecting adulterants would certainly be increased if the two kinds of oil were mixed, but he did not think this was done to any extent. He quite agreed with Mr. Hehner's remarks as to the value of the hexabromide test, and also with the suggestion as to the influence of small quantities of impurities on polymerisation. The oil extracted with petroleum ether polymerised much less readily than oil prepared by expression in the ordinary way. The Chinese oils, however, did vary appreciably in their polymerising power, so that heat tests based upon comparison with a single standard oil must be quite useless for indicating such small amounts of adulteration as were sometimes reported. The iodine test discovered by Mr. Jenkins was an excellent supplementary test. With regard to Mr. Bolton's question, he should not like to say positively that no genuine sample yielded a lower value than those he had given, but he thought that the figures he had recorded might be regarded as representing the limits which in the present state of knowledge it would be safe to adopt. He had met with lower iodine values, but was not sufficiently certain of the origin of the samples to include them.

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THE ESTIMATION OF MANGANESE BY THE BISMUTHATE METHOD.

By H. F. V. LITTLE, A.R.C.S., B.Sc.

(*Read at the Meeting, November* 6, 1912.)

THE bismuthate method for the estimation of manganese was originated by Schneider (*Dingl. poly. J.*, 1888, **269**, 224), and improved by Reddrop and Ramage (*J. Chem. Soc.*, 1895, **67**, 268); later it was modified by Ibbotson and Brearley (*Chem. News*, 1900, **82**, 269; 1901, **84**, 247, 302; 1902, **85**, 59), and by Blair (*J. Amer. Chem. Soc.*, 1904, **26**, 793). The method consists in oxidising the manganese—present as manganous salt—in a cold solution free from chloride, and containing 25 per cent. by volume of nitric acid of sp. gr. 1·42, to the state of permanganate by agitation with an excess of sodium bismuthate. The solution is filtered through asbestos and titrated. Reddrop and Ramage added a slight excess of $\frac{N}{10}$ hydrogen peroxide, and titrated back with $\frac{N}{10}$ permanganate; but since in the presence of any appreciable quantity of ferric salt the use of peroxide is attended by an error proportional to the excess of reagent used, Ibbotson and Brearley employ ferrous sulphate. Blair does the same, and only differs from the others in the strengths of the standard solutions he employs.

According to the writer's experience, there is no advantage gained by using reagents of other than decinormal concentration, and he prefers to carry out the method as recommended by Ibbotson and Brearley, and described in Arnold and Ibbotson's "Steel Works Analysis," diluting the oxidised solution, however, before filtration with an equal volume of 3 per cent. nitric acid. The object of the present note is to record a number of observations on the degree of accuracy of the method, which has been adversely commented upon by Brinton (*J. Ind. and Eng. Chem.*, 1911, **3**, 237, 376). According to this author, if the values of the standard solutions employed are deduced in the usual manner and calculated to manganese, the results are appreciably low, and the only way to obtain correct results is to determine an empirical factor by standardisation against a known amount of manganese.

So far as the estimation of small amounts of manganese is concerned, Brinton's statement has been refuted by Hillebrand and Blum (*J. Ind. and Eng. Chem.*, 1911, **3**, 374), and a number of experiments have convinced the writer that the method gives admirable results when large percentages of manganese are concerned and the theoretical factor is employed. Firstly, evidence is supplied by the results obtained by E. Cahen and the writer (ANALYST, 1911, **36**, 52) in determining the manganese in a number of ferros and pyrolusites by four different methods, for the theoretical factor was employed in working out the bismuthate results. Secondly, the test analyses given in Table I. lead to the same conclusion.

The results given in Tables I. and II. were obtained as follows: An approximately $\frac{N}{10}$ permanganate solution was prepared from twice recrystallised potassium permanganate and distilled water, measured portions were reduced in nitric acid solution by a slight excess of concentrated hydrogen peroxide, and the oxidation of the solution with bismuthate effected as usual. A measured excess of approximately

$\frac{N}{10}$ ferrous sulphate or hydrogen peroxide was added, and the excess titrated with the original permanganate solution, against which the ferrous salt and peroxide were checked. In each experiment marked with an asterisk the oxidised solution was filtered into an empty flask, and the titration subsequently performed. Low results are thus obtained. In the other experiments the oxidised solution was filtered directly into the ferrous salt or peroxide. The results are excellent.

TABLE I.

Manganese used in each experiment = 25 c.c. $KMnO_4$ solution.
25 c.c. $FeSO_4$ solution = 30·78 c.c. $KMnO_4$ solution.

$FeSO_4$. c.c. used.	$KMnO_4$. c.c. used.	Manganese found (in c.c. Original Solution).	Error.
25	5·83	24·95	−0·05
25	5·83	24·95	−0·05
25	5·77	25·01	+0·01
*25	5·88	24·90	−0·10
*25	5·92	24·86	−0·14
35	18·06	25·03	+0·03

TABLE II.

Manganese used in each experiment = 25 c.c. $KMnO_4$ solution.
25 c.c. H_2O_2 solution = 27·97 c.c. $KMnO_4$ solution.

H_2O_2. c.c. used.	$KMnO_4$. c.c. used.	Manganese found (in c.c. Original Solution).	Error.
25	2·95	25·02	+0·02
25	3·00	24·97	−0·03
25	3·00	24·97	−0·03
35	14·17	24·99	−0·01
35	14·18	24·98	−0·02
*25	3·08	24·89	−0·11
*25	3·10	24·87	−0·13

TABLE III.

$KMnO_4$ used in titrations = 0·10174Mn.
1 c.c. = 0·001119 grm. Mn.

Excess of $FeSO_4$ over $KMnO_4$.		manganese.		Error.
		Found.	Taken.	
		Iron absent.		
10·05 c.c. $KMnO_4$	...	0·01125	0·01128	−0·03 mgrm.
10·00 " "	...	0·01119	0·01128	−0·09 "
10·05 " "	...	0·01125	0·01128	−0·03 "
		Iron present.		
10·33 c.c. $KMnO_4$	...	0·01125	0·01128	−0·03 mgrm.
10·30 " "	...	0·01122	0·01128	−0·06 "
3·25 " "	...	0·00445	0·00450	−0·05 "
3·27 " "	...	0·00447	0·00450	−0·03 "

In the experiments on smaller amounts of manganese, recorded in Table III., the use of hydrogen peroxide was discontinued. Known quantities of manganese were obtained by reducing a weighed amount of pure potassium permanganate, diluting to a known volume, and taking measured amounts of this solution. The approximately $\frac{N}{10}$ permanganate used in the titrations was standardised against pure sodium oxalate, and the ferrous sulphate against the permanganate. In those experiments indicated as having been performed with iron present, sufficient ferric nitrate was added to contain 1 grm. of iron, and in each case under "manganese found" a correction has been introduced (0·00031) for the manganese present in the ferric nitrate and determined by a blank experiment.

The results are a trifle low—on an average 0·00005 grm.—which corresponds to only 1 drop of $\frac{N}{10}$ permanganate.* With small quantities of manganese, it is a matter of indifference whether the ferrous sulphate is added after filtration or the solution filtered into an empty flask.

The error in these latter experiments only represents 0·005 per cent. Mn, taking 1 grm. of alloy, and duplicate experiments in ordinary working do not agree any closer than this. For example, three estimations carried out at the same time on the same manganese steel gave the writer 0·764, 0·756, 0·757 per cent. Mn respectively, and other alloys gave 0·259, 0·260, 0·271 per cent. Mn, 0·245, 0·236, 0·243 per cent. Mn, and 0·210, 0·203, 0·206 per cent. Mn.

It should be mentioned that the writer has on several occasions encountered the curious experience which led Ramage (*Chem. News*, 1901, **84**, 269) to reject the use of ferrous sulphate, and is at a loss to account for it. Neither Ibbotson and Brearley nor Blair refer to this matter.

In the opinion of the writer, the bismuthate method is the neatest and most accurate of the volumetric methods that have been proposed for the estimation of manganese.

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The President writes as follows: The bismuthate method of estimating manganese has been in use in my laboratory for seventeen years, chiefly in connection with the analysis of steel and iron. It is certainly the neatest method I am acquainted with, one of the most rapid, and, when carefully carried out, is very accurate. We use Ibbotson and Brearley's modification. The following results, in which the same steels were analysed by the bismuthate process and by Ford's volumetric method, show how closely the two methods agree:

		Manganese	
	Carbon.	Ford's Method.	Bismuthate Method.
Steel casting	0·28	1·508	1·500 1·503
Tyre	0·41	1·185	1·191 1·183

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* This is probably due to the presence of a little water (about 0·4 per cent.) in the recrystallised permanganate employed (Blum, *J. Amer. Chem. Soc.*, 1912, **34**, 1379). Blum's study of the bismuthate method appeared some time after this paper was written.

		Manganese.	
	Carbon.	Ford's Method.	Bismuthate Method.
Forging	0·27	1·165	1·171
			1·170
Rail	0·38	0·993	0·992
		0·996	0·993
		1·005 1·003 *	0·995*
Crank axle	0·20	0·485	0·490
		0·484	
		0·488*	0·480*
" "	0·27	0·834	0·844
			0·841

The results marked with an asterisk (*) were obtained by Mr. J. H. B. Jenkins, who analysed the samples in duplicate with me.

* * * * *

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Detection of Adulteration by Colloidal Chemical Methods. E. Marriage. (*Zeitsch. Chem. Ind. Kolloide*, 1912, **11**, 1-5; through *Chem. Zentralbl.*, 1912, II., 1401.)—For the detection of cheap fruit-jelly, such as apple and gooseberry, and also of agar-agar in more expensive jams, a little of the sample is mixed with potassium iodide, and after cooling is covered with lead nitrate solution. From the appearance of the layers of lead iodide in the test-tube it is possible to draw conclusions as to the purity of the jam. Or a drop of the jam, to which 5 per cent. of potassium iodide has been added, is placed in a cell of 18 mm. diameter on a warmed object glass, and covered with a cover-glass of 16 mm. diameter. After cooling, the cell is filled up with 20 per cent. lead nitrate solution, and sealed with a large cover-glass, and the forms assumed by its contents examined under the microscope after the lapse of twelve to twenty-four hours. C. A. M.

Detection of Aniline Dyes and Turmeric in Mustard. E. Sievers. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 393-394.)—Although the method described by Bohrisch for the detection of turmeric (ANALYST, 1904, **29**, 372) is reliable, the twelve hours' extraction with alcohol renders it tedious, and the author, therefore, recommends the following method of procedure: A few grms. of the mustard are moistened with a drop or two of alcohol in a test-tube, and about 10 c.c. of ether are then added. The mixture is shaken, allowed to settle, and the ethereal layer is poured on a strip of filter-paper, and tested in the usual way for boric acid with turmeric. Bohrisch's process for the detection of aniline dyes (*loc. cit.*) is simple and trustworthy. W. P. S.

Detection of Minute Traces of Boric Acid by Means of Tincture of Mimosa Flowers. L. Robin. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1., 429-432.)—Tincture of mimosa flowers is a more sensitive reagent than turmeric for the detection of boric acid. The reagent is prepared by digesting the flowers with 20 parts of 95 per cent. alcohol for ten minutes at the temperature of the boiling water bath, allowing to cool, and filtering. Since organic acids interfere, it is first necessary to incinerate such materials as milk, wines, etc. The liquid to be tested is evaporated in a 2-inch porcelain, flat-bottomed dish, and the residue is burnt and moistened with a few drops of water. A few (2 to 4) drops of the reagent are added, and then 5 per cent. hydrochloric acid drop by drop until the liquid loses its yellow colour. On evaporation to dryness, the residue is citron yellow if boric acid is absent, and its colour is unchanged on treatment with ammonia. In presence of boric acid the residue has a yellowish-brown colour, which changes to red on moistening with dilute (1 : 3) ammonia. A distinct pink is shown by less than 0·0005 mgrm. of boric acid. This test shows that boric acid is not merely an occasional, but a normal constituent of wines, and that it is present in minute traces in many samples of genuine milk. G. C. J.

Detection and Estimation of Formic Acid in Syrups. H. Kreis. (*Mitt. Lebensmitteluntere. u. Hyg.*, 1912, 3, 205-210; through *Chem. Zentralbl.*, 1912, II., 1312-1313.)—The author criticises the official Swiss method, according to which 100 grms. of the substance are acidified with sulphuric acid and distilled until at least 100 c.c. of distillate are collected. According to him, it is not possible to ascertain qualitatively whether, in the case of an acid saccharine liquid, formic acid has been added or not; this can only be determined by a quantitative estimation of the formic acid, bearing in mind that formic acid may be produced in the course of the operation. The quantity of this latter depends on the manner of heating, the concentration of the liquid under distillation, and its acidity. The prescribed addition of dilute sulphuric acid should be omitted. If from 100 grms. of syrup, without the addition of acid, about 1 litre of distillate be driven over with a current of steam in the water-bath, it may safely be concluded that a quantity of formic acid exceeding 5 mgrms. cannot be due to the decomposition of the sugar, and that formic acid was originally present. The distillate obtained should be neutralised with calcium carbonate, being boiled under a reflux condenser with 1 to 2 grms. of finest precipitated chalk for ten minutes before it is evaporated down. J. F. B.

Fractional Saponification of Fats. A. J. J. Vandevelde. (*Ann. Falsif.*, 1912, 5, 417-421.)—Results of an investigation regarding the effect of partial saponification of fats are recorded, statements having been made that such treatment alters considerably the character of certain fats which are employed as adulterants of butter. Coconut oil, lard, and butter fat were saponified with one-half the quantities of sodium hydroxide required for their complete saponification; the unsaponified fats were then separated, an examination being also made of the insoluble non-volatile acids obtained from the soaps. The chief differences were noted in the mean molecular weights of the insoluble non-volatile fatty acids, particularly in the case of lard and butter fat. The molecular weights found were as follows:

	Fatty Acids from Original Fat.	Fatty Acids from the Non-saponified Portion.	Fatty Acids from the Saponified Portion.
Coconut oil ...	208	211	214
Lard	274	273	240
Butter fat ...	259	260	235

In the case of coconut oil, the saponification value was diminished from 271 in the original oil to 254 in the unsaponified portion, and from 236 to 219 in the case of butter fat, whilst the values for lard remained practically constant. The refractive indices and critical temperatures of solution were also lowered. W. P. S.

Experiments on the Value of Ordinary and Treated Sawdust for Animal Nutrition. F. Honcamp, B. Gschwender, H. Müllner, and M. Reich. (*Landwirths. Vers.-Stat.*, 1912, **78**, 87-114.)—An account is given of previous experiments by various authors, all of which tend to show that the fodder value of brushwood, sawdust, and wood-meal, whether raw or treated in various ways—*e.g.*, by fermentation, etc.—is extremely small. It is true that Lehmann had found that the fine subdivision of the wood, as in the case of wood-meal, slightly increased the digestibility of its components, but for practical purposes this effect was negligible. Pfeiffer, moreover, had shown that sawdust, besides affording no nutrition itself, also lowered the degree of utilisation of other fodder stuffs administered simultaneously. The subject has recently been reopened by the use of a product derived from sawdust by hydrolysis with sulphurous acid under pressure, with the formation of 20 to 25 per cent. of sugar. This product, obtained mainly from coniferous wood, occurs in the form of a brown meal, which is readily reduced to a fine dust by rubbing. It is utilised in admixture with molasses, for which it forms a solid vehicle, possessing certain physical advantages. The authors' experiments were carried out upon sheep with the raw sawdust and with the treated product derived from it. These were administered in admixture with a standard ration composed of hay, cottonseed cake, and brewers' grains molasses.

Proximate analyses of the two wood materials, calculated on the dry substance, gave the following results:

	Crude Protein.	Non-nitrogenous Extract.	Fat.	Crude Fibre.	Ash.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Raw sawdust ...	0·88	28·63	1·25	68·64	0·60
Treated sawdust	0·67	40·30	1·00	57·33	0·70

So far as the raw sawdust was concerned, the results of the present trials confirmed the observations of Pfeiffer. Not only was it without appreciable nutrient value in itself, but in certain directions it unfavourably affected the assimilation of the other components of the ration. The results obtained with the treated sawdust were slightly better in so far as the non-nitrogenous extract and the fat were concerned. The meal showed, on the dry substance, 26·20 and 0·73 per cent. respectively of these constituents assimilated. The quantity of fat introduced by the wood-meal is so small that its value is negligible. In the case of the non-nitrogenous extract, the improvement may be attributed to the saccharification of the wood fibre by the chemical treatment. Against this positive nutritive value must be set the fact that not only are the protein and fibre of the modified wood-meal totally unassimilable themselves, but the inclusion of this meal in the diet decreases the assimilation of the protein and fibre of the other constituents of the fodder. A further series of trials was made with a commercial fodder composed of the treated wood-meal and molasses. After correcting the results for the value of the molasses contained in the preparation, the conclusions arrived at in the case of the wood-meal itself were again confirmed. Thus the treated sawdust cannot be regarded as a satisfactory adjunct to molasses fodder, and more suitable vehicles must be sought for.

J. F. B.

Determination of Water in Molasses. C. C. Roberts. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 8, 53-37.)—Various methods of drying molasses for the estimation of moisture have been tested by the author. The direct drying of undiluted molasses in the air-oven at 98° to 99° C. gives low results owing to occlusion of moisture, and after a time the sample begins to gain in weight. The same remark applies when the sample is dried in a current of hydrogen. Drying the undiluted sample in air at 98° C. can only be performed when particular care is taken to place the sample drop by drop on the dish. Drying the diluted molasses on pumice is tedious, and yields low results. The author prefers to dry the diluted sample on a filter-paper coil, such as is used for the estimation of fat in milk by Adams' process. The water can be determined practically as exactly as in hydrogen and in a shorter time than by other methods. The sample is diluted to a uniform strength corresponding to about 30 per cent. of molasses. About 7 grms. of the solution, weighed by difference, are distributed as evenly as possible upon a dried coil of filter-paper, specially prepared for Adams' method, of dimensions 65 × 565 mm. The coil is dried for three hours in a water-oven, cooled and weighed. The loss is calculated as total water, including water of hydration of the sugars. If only the actual moisture is required, the sample must be dried at 70° C. to constant weight.

J. F. B.

Polarimetric Estimation of Banana Starch. G. Baumert. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 449-452.)—The methods described by Lintner (Analyst, 1897, **32**, 179) and Ewers (Analyst, 1908, **33**, 101, 481) may be employed for the estimation of this starch. Under the conditions of the former method banana starch has a specific rotation of +209·8°, whilst in Ewers' method this value is +196·4°. The finely divided banana substance should be extracted successively with cold water, alcohol, and ether before the starch is estimated. (See also Analyst, 1911, **36**, 588.)

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Substitute for the Petri Dish for Cultivating Fungi and for Biological Analysis. P. Lindner. (*Wochenschr. f. Brau.*, 1912, 29, 589-590.)—The ordinary Petri dish presents certain difficulties in manipulation, particularly as regards infection from the air at the time of inoculation or when opening the dish to take off sub-cultures or specimens for examination. The vessels known as Lindner's fungus-culture glasses are far more suitable for general purposes than Petri dishes. These glasses resemble in shape the inverted specimen jars, consisting of a round-ended cylinder with constricted neck, terminating in an enlarged foot. They are constructed of thin glass and are without stoppers, the opening in the foot being closed by a plug of cotton-wool. They are practically incapable of becoming infected from the air if properly manipulated, and the author has taken as many as sixty sub-sowings from one culture without infection. The walls are coated with an extremely thin layer of solid culture medium by rolling them round whilst the medium is fluid. The layer of medium and the glass both being extremely thin, the colonies are easily inspected. Contrary to the Petri dish, it is the underside of the colony which is seen in these glasses, and the peculiar ring structure of the growth is visible unimpeded by surface vegetation. Further, by binding a sheet of gaslight photographic paper around the cylinder, and exposing it to the light, a direct photographic record of the culture at any stage is easily obtained. J. F. B.

Estimation of Fat and Lipoids in Blood, and Notes on the So-called Lipolysis. L. Berczeller. (*Biochem. Zeitsch.*, 1912, 44, 193-200.)—Results of experiments carried out by the author confirm the statement made by Shimidzu (*cf.* ANALYST, 1910, 35, 527), that more fat is obtained by extracting the blood with alcohol, and saponifying this extract, than is found by saponifying the blood directly according to the Kumagawa-Suto method. It is recommended that 50 c.c. of blood be added drop by drop to 400 c.c. of alcohol; the mixture is then boiled for five hours, filtered while hot, and the precipitate washed with alcohol and ether. The filtrate and washings are evaporated to about 100 c.c., and then saponified as described by Kumagawa and Suto (ANALYST, 1908, 33, 362). Horse's blood yielded from 0·3 to 0·4 per cent., and dog's blood from 0·5 to 0·6 per cent., of fat by this method, results which were about 0·1 per cent. higher than those obtained by the direct saponification method. The loss of fat (lipolysis) which has been observed when blood is aerated is probably due to the formation of compounds of the fat with other constituents of the blood; these compounds are soluble in alcohol, but not in ether. Alterations in the structure of the lipoids may also take place. It does not seem probable that the loss is due to the action of bacteria. W. P. S.

Polarimetric Estimation of Glucosamine in Ovomucoid and Pseudomucin. C. Neuberg and O. Schewket. (*Biochem. Zeitsch.*, 1912, 44, 491-494.)—A method which has been described by Neuberg and Ishida (ANALYST, 1912, 145) for the estimation of sugars in the presence of amino acids, peptones, etc., may also be employed for the estimation of glucosamine. It depends on the fact that amino acids, peptones, and albumoses, are precipitated by mercuric acetate and phosphotungstic acid, whilst sugars and glucosamine in neutral solution are not precipitated by these reagents. One grm. of ovomucoid is hydrolysed by boiling for three hours with 50 c.c. of 7 per cent. hydrochloric acid, the solution is then evaporated, and the residue is dissolved in 13 c.c. of water. A 25 per cent. mercuric acetate solution is now added in quantity sufficient to make the total volume 25 c.c. After filtration 12·5 c.c. of the filtrate are treated with 2·5 c.c. of a concentrated phosphotungstic acid solution, the mixture is again filtered, and the filtrate examined in the polariscope. In the case of pseudomucin the hydrolysis with hydrochloric acid should be continued for thirty hours. Ovomucoid was found to contain 24 per cent., and pseudomucin 36 per cent., of glucosamine. W. P. S.

Detection of Combined Glycuronic Acid in Normal Urine. C. Neuberg and O. Schewket. (*Biochem. Zeitsch.*, 1912, **44**, 502-504.)—The naphthoresorcinol test described by Tollens (Analyst, 1908, **33**, 326) is a general reaction for carboxylic acids, and the orcinol test for glycuronic acid also gives a reaction with pentosans. Both these reactions, however, may be used for the detection of combined glycuronic acid if they are applied to the ethereal extract of a urine, and not to the latter directly. The test is carried out as follows: Ten c.c. of the urine are acidified with 2 c.c. of dilute sulphuric acid, 10 c.c. of alcohol are added, and the mixture is shaken out with 20 c.c. of ether. The ethereal extract is washed with water, filtered, and evaporated after the addition of a small quantity of water. The residual aqueous solution is then divided into two portions, to which the tests are applied. The urine to be tested should be as fresh as possible; when urine is kept, the combined glycuronic acid is decomposed, with the formation of free glycuronic acid which is not soluble in ether. W. P. S.

The Indole Reaction. H. Zipfel. (*Zentralbl. Bakt. Parasit.*, 1912, **64**, 65-80; through *Chem. Zentralbl.*, 1912, II., 1497.)—The possibility of the production of indole by bacteria depends on the presence of the tryptophane group (indole-α-aminopropionic acid) in the nutrient medium. The author recommends the use of a medium prepared by dissolving 5 grms. of asparagin, 5 grms. of ammonium lactate, 2 grms. of dipotassium phosphate, 9·2 grms. of magnesium sulphate, and from 0·1 to 0·5 grm. of tryptophane, in 1 litre of water. This solution has the advantage over peptone solution in being free from colour; it is also constant in composition. Any of the ordinary tests may be employed for detecting the formation of indole by bacteria in a tryptophane medium, but Böhme's *p*-dimethylaminobenzaldehyde reaction gives the best quantitative results. W. P. S.

Occurrence of Mercury in the Hair of Persons who have received Subcutaneous Doses of Mercury Compounds. Detection of Minute Quantities of Mercury. C. Strzyzowski. (*Chem. Zeit.*, 1912, **36**, 1237-1239.)—By means of the following method the author has detected traces of mercury in the hair of persons who had previously been subjected to treatment with mercury compounds. The method is capable of detecting the presence of 1 part of mercury in about 90,000,000 parts of substance. From 2 to 10 grms. of the hair are washed successively with ether, alcohol, and hot water, and are then digested with hydrochloric acid, small quantities of potassium permanganate being added from time to time until the substance has dissolved. The solution is filtered, evaporated partially, and then treated with hydrogen sulphide; after the lapse of twenty-four hours, the precipitate, consisting of sulphur and traces of mercury sulphide, is collected on a filter, then dissolved in a small quantity of hydrochloric acid and potassium chlorate, the solution is filtered, and the filtrate is evaporated to about 1·5 c.c. This residual solution is diluted, filtered, and a few small strips of copper-foil are added to the filtrate, which is then boiled for a few minutes; the copper-foil is now removed, dried, and transferred to a tube, the upper end of which is then drawn out to a capillary. After the air has been exhausted from the tube (while the lower part of the tube is heated to about 130° C.) the capillary is sealed, and the tube is heated over a flame in order to volatilise the mercury from the foil into the capillary. The latter is then examined under the microscope, when minute globules of mercury will be observed. These globules may be rinsed with alcohol from the capillary, and treated on a microscope slide with a trace of iodine. A characteristic action of the iodine on the globules may be observed under the microscope. W. P. S.

Toxicity of Mushrooms: Their Hæmolytic Action. J. Parisot and Vernier. (*Compt. rend.*, 1912, **155**, 620-623.)—The symptoms observed in cases of poisoning by mushrooms point to hæmolysis as the cause of death. This has been confirmed, both *in vitro* and *in vivo*, by experiments made with extracts of fungi in sodium chloride solutions containing 9 grms. of salt per litre. Examination of the blood of animals killed by the injection of these extracts showed a cherry-red coloration of the serum (*hæmoglobinæmia*), and the globules were hæmolysed in saline solutions. The *Amanita phalloides*, Fr., was found to possess the most powerful hæmolytic action, but all poisonous species had an intense, though less marked, action. Moreover, many of the edible species also exert a destructive action on the blood, notably *Amanita rubescens*, Pers., *Hydnum repandum*, Lin., *Tricholomia nudum*, Fr., *Laccaria laccata*, Be. and Br., *Craterellus cornucopioides*, Fr., etc. Certain species which do not cause hæmolysis when young and fresh will develop this property as they get older. Generally the hæmolytic action is attenuated by heating, but in certain cases a prolonged period of heating at a high temperature is not sufficient to destroy it entirely. The authors' experiments show that the hæmolytic action is due to a glucoside. The activity of the glucoside is very considerably weakened and retarded by certain substances, such as milk, and particularly cholesterol (yolk of egg). When the hæmolytic action of the fungus extracts had been destroyed by heating, it was found that their toxic action was either very much attenuated or eliminated. When subjected to the ordinary operations of cooking, the mushrooms frequently retain their hæmolytic activity to a certain extent, and it is recommended that such fungi should be cooked for a prolonged time at a high temperature, preferably with the use of oil, in order to effect the complete hydrolysis of the hæmolytic glucoside. J. F. B.

Estimation of Oxalic Acid in Vegetable Substances. A. Gregoire and E. Carpiaux. (*Bull. Soc. Chim. Belg.*, 1912, **26**, 431-434.)—Oxalic acid, in the form of its calcium, potassium, and sodium salts, is found widely distributed in the vegetable kingdom, and to a much less extent in the animal kingdom. At the same time this acid is but rarely estimated in the ordinary course of analysis of vegetable foodstuffs, and the authors therefore describe a method for its estimation, this method having been applied particularly to such substances as sesame cakes and other products, which also contain large quantities of phosphates, calcium salts, and proteins. Five grms. of the finely powdered, fat-free cake are heated on a water-bath for one hour with 20 c.c. of 4 per cent. hydrochloric acid; a quantity of sodium sulphate sufficient to precipitate the calcium salts present is then added, and after cooling the mixture is mixed with about 100 c.c. of 94 per cent. alcohol. The precipitate is collected on a filter, washed with alcohol, a slight excess of ammonia is added to the filtrate, the alcohol is removed by evaporation, and the solution is acidified with hydrochloric acid and filtered. Calcium acetate is added to the filtrate, the calcium oxalate is collected on a filter, washed, dissolved in hydrochloric acid, and the solution evaporated nearly to dryness. This residue is acidified with a few drops of 25 per cent. sulphuric acid, and sufficient anhydrous sodium sulphate is added to form a dry mass, which is then extracted six successive times with ether. The ethereal extracts are rendered ammoniacal, evaporated, the residue is dissolved in water, and the oxalic acid is precipitated with calcium acetate, collected, ignited, and weighed. A sample of sesame cake examined yielded a quantity of oxalic acid, corresponding with 2·94 per cent. of crystallised calcium oxalate ($CaC_2O_4.H_2O$).

W. P. S.

Estimation of the Activity of Commercial Rennet. W. van Dam. (*Landw. Vers.-Stat.*, 1912, **78**, 133-163; through *Chem. Zentralbl.*, 1912, II., 1313-1314.)—The author has observed that in presence of a low hydrogen ion concentration ($2·37 \times 10^{-6} N$) the digestive activity of rennet towards paracasein runs exactly parallel to its coagulating activity. He has based on this an accurate method for establishing the activity of a normal rennet, and he proposes as a standard 1:100,000 rennet powder, a preparation which yields digestion products, under the conditions prescribed, which are equivalent to 26·5 c.c. of $\frac{N}{10}$ acid in Kjeldahl's nitrogen estimation. The digestion experiment is carried out as follows: Into a 100 c.c. flask is weighed as much purified casein as corresponds to 0·8 grm. of anhydrous substance (Kjeldahl factor 6·37). To this are added 20 c.c. *N*-acetic acid, 10 c.c. *N*-sodium hydroxide, and 70 c.c. of boiled water, whereby the above-mentioned concentration of hydrogen ions is established. The flask is slowly rotated in the thermostat at 30° C. After settling, 1 c.c. of a 1 per cent. solution of the rennet powder is added. A control test is also set up containing 1 c.c. of the rennet extract previously heated at 90° to 100° C. for ten minutes. The digestion is continued for twenty-four hours at 30° C.; 75 c.c. are then filtered off for the Kjeldahl estimation, and the number of c.c. of $\frac{N}{10}$ acid neutralised represents the digestion value. By means of the square root law, according to which the quantity of casein digested is proportional to the square root of the enzyme concentration, it is calculated what concentration of the rennet powder must be taken to give a result approximating to 26·5 c.c. of acid. This solution must then be compared with one or more calf-stomach infusions, in order to verify the parallelism of the digestive and coagulating functions. The neutralised infusions of calf-stomach are diluted until their coagulating power is approximately equal to that of the rennet powder solution, and the digestive activity of these infusions is then estimated. If the digestion and coagulation are found to be sufficiently parallel, the solution of rennet powder is again tested for its digestive activity, and, provided the result does not differ by more than 2 c.c. from 26·5 c.c., the strength of the powder may be calculated by the square root law on the basis of 26·5 c.c. for a standard powder of 1 : 100,000 in 1 per cent. solution for 75 c.c. of digested products. J. F. B.

Colorimetric Method for the Estimation of Tryptophane, and the Quantity of Tryptophane in Horny Tissue and Other Proteins. H. Fasal. (*Biochem. Zeitsch.*, 1912, **44**, 392-401.)—The method is based on the violet coloration which is produced when tryptophane, or a substance containing it, is treated with concentrated sulphuric acid and glyoxylic acid. The coloration obtained is compared with that produced by known quantities of pure tryptophane. The author finds that the use of glyoxylic acid in the test is preferable to that of other aldehydes. In testing horn substance, horny epidermis, etc., the material was previously subjected to peptic and pancreatic digestion, and extraction with alcohol and ether. The following quantities of tryptophane were found in various materials: Horny epidermis, 0·30 per cent.; horn (ox), 0·17 per cent.; human nails, trace; hair (white, blonde, and dark), none; casein, 0·65 per cent.; edestin, 0·38 per cent.; lact-albumin, 3·07 per cent. W. P. S.

ORGANIC ANALYSIS.

New Method for Estimation of Alchohol. G. B. Frankforter and F. C. Frary. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 22, 87-127.)—When sufficient potassium fluoride is added to aqueous alcohol, the liquid separates into two layers. If now water is added gradually, with shaking between each addition, a point is reached when the solution again appears homogenous. The end point can be determined very closely if a little solid spirit blue be present, since this makes a very thin film of alcohol easily visible. The analytical method consists in transferring some pure, anhydrous potassium fluoride to a weighed flask, weighing, adding some of the aqueous alcohol to be tested, reweighing, cooling to about 25° C., and adding water a drop at a time until the solution becomes homogenous. A final weighing of the flask is then made, and the alcoholic strength of its contents, and thus of the sample, calculated by reference to Table I. Reference to the table is necessary before commencing an analysis. For example, if a spirit believed to contain from 40 to 50 per cent. of alcohol is to be analysed, the table shows that as much as 12 grms. of potassium fluoride may be necessary to insure separation into two layers if 100 grms. of spirit is taken for the test. If the quantities taken are exactly 12 grms. and 100 grms., and if 20 grms. of water is necessary to render the solution homogenous, then the final solution contains 12 grms. of potassium fluoride in 120 grms. solvent, or 10 grms. per 100 grms. solvent. By reference to the table, it is seen that this corresponds to 44 per cent. alcohol. This is the strength of the alcohol at the end of the experiment after the addition of 20 per cent. of water, and therefore the strength of the original spirit was 52·8 per cent. Potassium fluoride does not salt out methyl alcohol, but the above method gives accurate results with methylated spirit between 15 and 30 per cent. total alcoholic strength. With stronger or weaker methylated spirit the method gives results which may be in error by as much as 1 per cent.

TABLE I.

Grms. per 100 *Grms. Solvent.*

KF.	C_2H_5OH.	KF.	C_2H_5OH.	KF.	C_2H_5OH.
60	2·82	40	6·32	20	23·4
59	2·92	39	6·65	19	25·1
58	3·02	38	7·01	18	26·9
57	3·13	37	7·40	17	28·8
56	3·24	36	7·83	16	30·8
55	3·36	35	8·30	15	32·9
54	3·48	34	8·81	14	35·0
53	3·61	33	9·37	13	37·2
52	3·75	32	9·98	12	39·4
51	3·90	31	10·64	11	41·7
50	4·06	30	11·36	10	44·0
49	4·23	29	12·14	9	46·4
48	4·41	28	12·99	8	49·0
47	4·60	27	13·92	7	51·9
46	4·80	26	14·94	6	55·1
45	5·01	25	16·07	5	58·7
44	5·24	24	17·33	4	62·8
43	5·48	23	18·75	3	67·5
42	5·74	22	20·26	2	78·6
41	6·02	21	21·80		

Potassium fluoride absorbs 62 per cent. of its weight of water against 32 per cent. taken up by lime, and, probably on account of its greater solubility, its dehydrating action is more rapid than that of most drying agents. It cannot, however, be used for the production of alcohol of a strength exceeding 97·5 per cent.

Though potassium fluoride is the most suitable substance for use in the analytical method above described, potassium carbonate may be used. Very small amounts of insoluble matter render the end point uncertain, and it is therefore better to use a filtered, concentrated solution of known strength. The best indicator is solid phenolphthaleïn, as potassium carbonate bleaches spirit blue. Table II. gives a guide as to suitable quantities for analysis and the means of calculating the results:

TABLE II.

Grms. per 100 *Grms. Solvent.*

K_2CO_3.	C_2H_5OH.	K_2CO_3.	C_2H_5OH.
45	5·0	22	17·8
44	5·2	21	18·9
43	5·4	20	20·0
42	5·7	19	21·1
41	6·0	18	22·3
40	6·3	17	23·5
39	6·6	16	24·7
38	7·0	15	26·0
37	7·4	14	27·2
36	7·8	13	28·5
35	8·3	12	29·8
34	8·8	11	31·1
33	9·3	10	32·5
32	9·9	9	34·0
31	10·5	8	35·7
30	11·2	7	37·5
29	11·8	6	39·6
28	12·5	5	42·0
27	13·2	4	44·7
26	14·0	3	48·3
25	14·9	2·5	50·3
24	15·8	2	52·6
23	16·8		

G. C. J.

Detection of Methyl Alcohol in Ethyl Alcohol and Spirits. A. Bono. (*Chem. Zeit.*, 1912, **36**, 1171.)—Twenty-five c.c. of spirits and 50 c.c. of water (or 10 c.c. of strong alcohol diluted to 75 c.c.) are distilled in a flask with a delivery tube passing to the bottom of a second flask containing 50 c.c. of a cold saturated solution of potassium bichromate with 60 c.c. of sulphuric acid per litre. The vapours issuing from this second flask pass through a condenser into a graduated cylinder. The first 25 c.c. of the distillate, in which formaldehyde can rarely be detected, are rejected, whilst the next 10 c.c. are tested for formaldehyde as follows: Ten drops of a 0·5 per cent. aqueous solution of phenylhydrazine hydrochloride, 1 drop of 0·5 per cent. sodium nitroprusside solution, and 10 drops of 10 per cent. sodium hydroxide solution, are added to 2 c.c. of this fraction, which in the presence of formaldehyde gives a blue coloration changing to green and then to yellowish-red. About 1 per cent. of methyl alcohol may thus be detected. C. A. M.

Analysis of Oil of Citronella. V. Boulez. (*Bull. Soc. Chim.*, 1912, **11**, 915-917.)—From 25 to 50 grms. of the oil are treated with 100 (or 200) grms. of a concentrated solution of sodium bisulphite prepared by saturating the ordinary commercial solution of sp. gr. 1·345 with sodium sulphite. The mixture is shaken until the citronellal has combined with the bisulphite, after which it is allowed to stand for two to three hours, then diluted with 100 (or 200) c.c. of water, and heated with occasional agitation for several hours on the water-bath beneath a reflux condenser. There should then be a sharp separation between the dissolved aldehydic part and the uncombined oily part of the sample. The latter is collected in a tared flask and weighed, and the difference between the result and the original weight of oil gives the amount of citronellal. The geraniol is then estimated in the uncombined portion by acetylation in the usual way. Four samples of citronella oil thus analysed gave the following percentage results:

	I.	II.	III.	IV.
Citronellal	18·00	19·00	58·00	63·00
Geraniol	44·28	45·59	36·16	35·56
Citronellal and geraniol expressed as geraniol ...	62·07	—	—	81·50

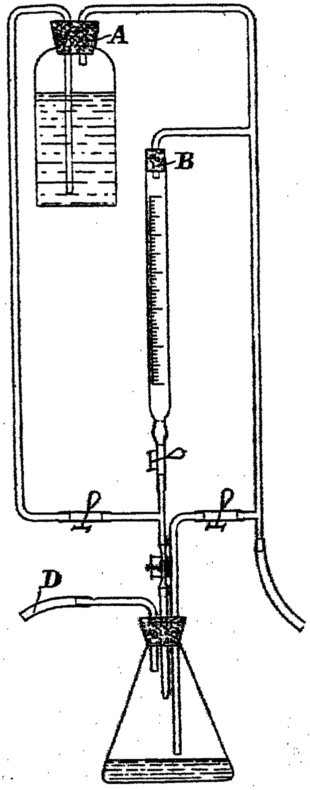
C. A. M.

Determination of Aldehydes. B. G. Feinberg. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 187-203.)—An investigation concerning the accuracy with which formaldehyde, benzaldehyde, salicylaldehyde, *p*-hydroxybenzaldehyde, anisaldehyde, and vanillin, may be estimated by methods depending on the use of bisulphite, neutral sulphite, or substituted hydrazines. The most accurate method for the estimation of benzaldehyde depends on the use of *p*-nitrophenylhydrazine. A convenient concentration of benzaldehyde is 0·25 grm. in 75 c.c. of 4 per cent. acetic acid. To this about twice the theoretical amount of the reagent is added, dissolved in 30 c.c. of 30 per cent. acetic acid. After five hours the precipitate is collected on a Gooch crucible, washed with 10 per cent. acetic acid, and dried at 105° to 110° C. Each gram of precipitate should correspond to 0·440 grm. benzaldehyde. Under the conditions described the benzaldehyde will be underestimated by 1·2 ± 0·7 per cent. A similar method gives good results (error ± 0·5 per cent.) with anisaldehyde and vanillin, but these aldehydes can be estimated with almost equal accuracy by means of *p*-bromphenylhydrazine, which is useless as a quantitative precipitant for benzaldehyde. The best results for salicyl aldehyde were obtained with *p*-nitrophenylhydrazine, but the error, though fairly constant, was large, namely $-5 \pm 0·5$ per cent. G. C. J.

Titration of Azo Dyestuffs by Means of Hydrosulphite. E. Grandmougin and E. Havas. (*Chem. Zeit.*, 1912, **36**, 1167-1169.)—A solution of an alkali hydrosulphite reacts with azo dyestuffs in accordance with the equation:

$$R.N:N.R' + 2Na_2S_2O_4 = RNH_2 + R'NH_2 + 4NaHSO_3.$$

The reagent is prepared by dissolving about 3 grms. of solid sodium hydrosulphite in a litre of recently boiled and cooled water, to which 5 c.c. of 30 per cent. sodium hydroxide solution has been added, and allowing the solution to stand for one to two hours. It should be used within four or five hours of its preparation. For the titration of the dyestuff the reagent is introduced into the flask, *A*, and the tube, *B*, of the apparatus shown in the figure, whilst 50 c.c. of a 0·1 per cent. solution of the dyestuff is placed in the lower flask, where it is diluted to 100 or 150 c.c., and treated with 5 c.c. of dilute hydrochloric acid (1 vol. of 36 per cent. acid to 10 vols. of water). The apparatus is then closed, and the air expelled from it by a current of gas, which is subsequently lit at a burner connected with *D*, after which the burette is filled and the dyestuff titrated. The method can not only be used in comparative estimations, but will yield absolute values by standardising the reagent upon a sample of a dyestuff of known purity. The amounts of hydrosulphite used are inversely proportional to the molecular weights of the anhydrous dyestuffs, as is shown by the following examples:



	C.C.	Molecular Weight.
Orange II.	28·0	350
Chrysoin	31·0	316
True Red (*Echtrot*) ...	24·5	400

In a series of estimations of five commercial samples of Orange II. the standard was prepared by twice recrystallising a sample of this dyestuff from a weak solution of sodium chloride, washing the crystals, and drying them at 130° to 140° C. until constant in weight. Taking this now to represent 100 per cent. of the pure dyestuff, the results obtained in comparison with the commercial samples showed percentages ranging from 75·2 to 89·3. So far the method has only been applied to acid mono-azo dyestuffs, which on reduction are sharply transformed into colourless products. Modifications will be necessary to adapt the method to the examination of other azo dyestuffs, such as poly-azo dyestuffs, which only dissolve with difficulty. The accuracy of the method has been controlled by precipitation of the acid dyestuffs with basic dyestuffs and by colorimetric comparisons. C. A. M.

Arsenic Xanthogenate and its Use in Analysis. N. Tarugi and F. Sorbini. (*Boll. Chim. Farm.*, 1912, 51, 361-370; through *Chem. Zentralbl.*, 1912, II., 1398-1399.)—Arsenic xanthogenate, $As(SCSOC_2H_5)_3$, is prepared by mixing a slightly alkaline solution of potassium arsenite with a solution of potassium xanthogenate, allowing the mixture to stand for fifteen minutes, acidifying it with acetic acid, washing the crystalline precipitate with water, and recrystallising it after drying over sulphuric acid. It melts at 94·8° C. (corr.), is completely insoluble in hot and cold water, and does not volatilise with steam. It is sparingly soluble in alcohol, more soluble in ether, benzene, carbon bisulphide, and chloroform, and soluble in hot glycerol, vegetable oils, vaseline, and lanoline. When heated with aniline it is quantitatively decomposed into diphenylthiourea and arsenic trisulphide. Cold mineral acids do not attack it, whilst cold concentrated solutions of caustic alkali dissolve it unchanged. The reaction with aniline may be used in the estimation of minute quantities of arsenic, the separated arsenic xanthogenate being heated on the water-bath with a few drops of aniline, and the residue of arsenic trisulphide heated at 150° C. until constant in weight. Quantitative results are obtained, but in estimating minute traces of arsenic it is preferable to extract the arsenic xanthogenate with chloroform and to evaporate the extract. Conversely the method may be used in the analysis of potassium xanthogenate. For a volumetric estimation 10 c.c. of an alkaline solution of arsenic trioxide of known strength are vigorously shaken with 5 c.c. of the potassium xanthogenate solution and allowed to stand for fifteen minutes, after which the liquid is acidified with acetic acid, diluted with water, vigorously shaken with 20 c.c. of benzene to dissolve the arsenic xanthogenate, and allowed to settle. The aqueous layer is now made up to 100 c.c., 10 c.c. thereof neutralised with sodium bicarbonate, and the excess of arsenic titrated with $\frac{N}{10}$ iodine solution, each c.c. of which is equivalent to 0·024 grm. of potassium xanthogenate.

Carbon bisulphide: This may be detected by means of an arsenite solution containing aniline and alcohol. The solution under examination is added to this reagent, or the air is made to bubble through it for some minutes; the liquid then neutralised with dilute sulphuric acid and diluted with water, and the precipitate freed from arsenic sulphide by recrystallisation. Glistening white crystals of α-diphenylthiourea (melting-point, 151° C.) are obtained:

$$CS_2 + 2C_6H_5NH_2 = H_2S + (C_6H_5NH)_2CS.$$

The reaction is not quantitative. A gravimetric method of estimating carbon bisulphide is to add the solution drop by drop to a solution of arsenic trioxide in potassium hydroxide solution containing alcohol, with continual cooling, or to pass the air to be tested through this reagent. After standing for fifteen minutes, the liquid is acidified with acetic acid, and allowed to stand for twelve hours, and the precipitate separated and weighed. Or carbon bisulphide may be estimated volumetrically with arsenite solution in an analogous manner to that used in estimating potassium xanthogenate. C. A. M.

Composition and Properties of Glycin Picrate and Separation of Glycin from Alanin. P. A. Levene and D. D. van Slyke. (*J. Biol. Chem.*, 1912, **12**, 285; through *Chem. Zentralbl.*, 1912, II., 1196.)—The formula of glycin picrate was found by analysis to be $(C_2H_5O_2N)_2.C_6H_3O_7N_3$ and not $C_2H_5O_2N.C_6H_3O_7N_3$. It melts at 199-200° C., decomposes at 202° C., and is very soluble in hot water, while water at 0° C. dissolves only 1·7 grms. per 100 c.c. For the separation of glycin and alanin the mixture is dissolved in 3 to 4 parts hot water, picric acid added in quantity less than sufficient to neutralise both bases, but more than enough for the glycin alone, and the glycin picrate allowed to crystallise out at 0° C. O. E. M.

Estimation of Glycerol in Fats. R. Willstätter and A. Madinaveitia. (*Ber.*, 1912, **45**, 2825-2828).—The method of Zeisel and Fanto, according to which the fat is saponified with alcoholic potash and the glycerol is afterwards converted into isopropyl iodide by the action of hydriodic acid, is generally considered to be the most accurate method for the estimation of glycerol. The authors now propose an improved method, whereby the fat is treated directly with hydriodic acid. About 0·2 grm. of the glyceride is weighed out into the decomposition flask of the Zeisel-Fanto apparatus and treated with 10 c.c. of hydriodic acid of sp. gr. 1·80. The mixture is heated to 100° to 115° C. (bath temperature) until reaction sets in, as shown by a strong separation of iodine and precipitation of the silver solution in the receiver. The temperature is maintained constant until the silver solution clears again (twenty to forty minutes) and the reaction is practically finished. Then the bath is raised to a temperature of 130° to 140° C., and heating is continued for at least an hour. This is not important in the case of triolein, but is necessary with tristearin. The isopropyl iodide distilled over is weighed as silver iodide. Results are satisfactory, *e.g.*, 9·87 per cent. of glycerol for tristearin, against 10·33 calculated, and 10·32 per cent. for triolein, against 10·41 calculated. If hydriodic acid of sp. gr. 1·70 be employed the results are too low, especially when larger quantities of fat are taken for analysis.

Note on the Acetyl Value of Fats.—As a measure of the hydroxy fatty acids and fat alcohols, Benedikt and Ulzer determine the acetyl value of the portion of saponified fat acid insoluble in water. Lewkowitsch (*J. Soc. Chem. Ind.*, 1897, **16**, 503) proposed instead to estimate the acetyl value of the fat direct without previous saponification. The authors have found that such treatment is not permissible, since acetic anhydride displaces the higher fatty acids in glycerides, with production of acetins. J. F. B.

Quantitative Estimation of Active Hydrogen in Organic Compounds by Magnesium-methyl Iodide. T. Zerewitinoff. (*Ber.*, 1912, **45**, 2384-2389.)—Hibbert (*Chem. Soc. Trans.*, 1912, **101**, 328) has stated that the lower alcohols of the fatty series, when treated with magnesium-methyl iodide in phenetole solution, yield considerably less than the theoretical quantity of methane. Working according to his standardised method for the estimation of hydroxyl groups or "active hydrogen" (ANALYST, 1911, **37**, 117) and using pyridine as the solvent, the author fails to confirm this statement and has obtained theoretical results from methyl, ethyl, propyl, and isobutyl alcohols. He has further extended the investigation of his method to the case of diamines, including representatives of the aliphatic, benzene, benzidene, stilbene, and naphthalene series. The estimations were carried out both in pyridine and anisole solutions. The results were concordant, and showed that substances containing two NH_2 groups react with two atoms of "active hydrogen" at the ordinary temperature, and with three atoms on heating at 85° C., but that the fourth atom does not react at all with magnesium-methyl iodide. Malonamide is an exception, reacting with four atoms on heating, but in the author's view one of these "active hydrogen" atoms is derived from the CH_2 group. The hydrocarbons of the indene and fluorene type in pyridine solution do not react at the ordinary temperature, but on heating at 85° C. for five minutes, a quantity of methane is obtained corresponding to one atom of "active hydrogen." The same applies for the hydrocarbons derived from fluorene by the substitution of a phenyl or naphthyl radical for one hydrogen in the CH_2 group; on the other hand, the carbinols derived from these latter hydrocarbons behave as tautomeric bodies, reacting only partially at the ordinary temperature and with only one "active hydrogen" atom on heating. J. F. B.

New Stability Test for Nitrocellulose Powders. S. A. Weirman. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 4, 157-159.)—The ability of a powder to take up and retain moisture is suggested as a measure of its stability, on the ground that this determines the amount of its hydrolytic decomposition. The sample, of which 10 grms. are sufficient, is weighed, dried for seven days in a sulphuric acid desiccator and weighed again; it is then exposed over water in a desiccator for forty-eight hours, and the gain in weight noted. The total range in weight divided by the surface area of the grain gives the actual "moisture range," values of which for different sizes of grain are comparable. The results are found to be parallel with those of the potassium-iodide starch test. O. E. M.

Exudation of Nitroglycerine from Dynamite. C. P. Beistle. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 4, 7.)—The dynamite is placed in a centrifugal machine, the cups of which describe a circle of 7-inch radius, and rotated for one minute at 600 revolutions per minute. The results obtained agreed with those of the 40° C. oven tests whereas the compression method gives results dependent upon the nature of the absorbent used. O. E. M.

Separation of Nitroglycerine from Nitro-Compounds. A. L. Hyde. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 4, 69-76.)—Complete fractional separation by two immiscible solvents, in one of which the nitroglycerine is more soluble than the nitro-compound, while in the other these conditions are reversed, could not be effected; but an empirical method, in which acetic acid and carbon disulphide were the solvents chosen, gave a definite fraction of the nitro-compound, nearly free from nitroglycerine, in the carbon disulphide. The proportion recovered was dependent on the particular nitro-compound under investigation. O. E. M.

Effect of the Nitrotoluenes on the Determination of Nitroglycerine by Means of the Nitrometer. C. P. Storm. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 4, 117-125.)—The nitric acid liberated from nitroglycerine by sulphuric acid in the nitrometer is taken up quantitatively by admixed mononitrotoluene until this is all converted into dinitrotoluene, if there is sufficient nitroglycerine. Hence, mixtures of these two substances containing less than 35·6 per cent. of nitroglycerine, yield no gas in the nitrometer; with more than this amount the error is 0·55 grm. of nitroglycerine per grm. of mononitrotoluene present. Pure dinitrotoluene and trinitrotoluene are without effect on the determination, but commercial liquid products cause an error proportionate to their content of mononitrotoluene. A liquid dinitrotoluene investigated caused an error of 0·0628 grm. nitroglycerine per grm., and therefore contained 11·36 per cent. of mononitrotoluene. An explosive containing 25 per cent. of nitroglycerine and 10 per cent. of this liquid dinitrotoluene would thus show an error of 0·628 per cent. in its nitroglycerine content. O. E. M.

Modified Form of Stability Test. H. C. P. Weber. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 4, 147-156.)—The sample, if necessary cut into pieces weighing about 0·2 grm., is placed in a test-tube partly immersed in a glycerol or oil bath, and the time required to produce explosion measured with a stop-watch. The temperatures chosen for the bath in this investigation were 200°, 180°, and 160° C.; for each sample at least three tests were made at each temperature, and a curve plotted from the average values and the temperatures. The curves fell into three groups for the samples tested, corresponding to their known classification into good, fair, and bad. Stable powders gave a pronounced bend, and a ratio of explosion periods at 200° and 160° C. of at least 2 : 9, while the ratio fell to 2 : 3 with unstable powders, and the points did not fit a smooth curve so well. Nitrocellulose gave a flatter curve, more nearly corresponding to the theoretical curve constructed on the assumption that the reaction velocity doubles for every 10° C. O. E. M.

Determination of Water in Petroleum and its Products. J. C. Allen and W. A. Jacobs. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 10, 17-23.)—A criticism of ten methods, all of which find use for the determination of water in petroleum or its products. The most exact is that depending on the volume of hydrogen evolved when the sample is treated with finely-cut sodium. The manipulation is identical with that of the well-known carbide method, the results of which are less accurate on account of the solubility of acetylene in oils. Of the other methods, the most accurate is direct distillation of a considerable quantity (200 grms.) of the sample. This method is accurate to within 0·03 per cent. if the distillation is pushed to 150° C., the water removed from the receiver by a micro-pipette and weighed, and the condenser cleaned with a tared plug of absorbent cotton, which has been previously wetted and squeezed as dry as possible. Ice-water should be fed to the condenser to obtain results of the accuracy stated. The other methods described, though useful for some purposes of works control, are only approximately exact, and some of them are only appliable to heavy oils and greases.
G. C. J.

Conditions for the Complete Hydrolysis of Proteins. D. D. van Slyke. (*J. Biol. Chem.*, 1912, 12, 295; through *Chem. Zentralbl.*, 1912, II., 1222.)—Various proteins were heated with 20 per cent. hydrochloric acid at 100° C., and also with 3 N. hydrochloric acid at 150° C., and the ammonia, total nitrogen, amino-nitrogen, and melanin determined in the reaction-product. The amino-nitrogen reaches a maximum, which is the same whether the heating is at 100° or 150° C., when the acid hydrolysis of a protein is complete. The author was unable to confirm the statement of Henriques and Gjaldbaek (*Zeit. physiol. Chem.*, 1910, 67, 8) that egg albumin, when heated with 20 per cent. hydrochloric acid at 100° C., gives only 90 per cent. of the amino-nitrogen obtainable by heating with 3 N. hydrochloric acid at 150° C.
O. E. M.

Rapid Estimation of Pyridine in the Presence of Ammonia in Gas-Water. O. Baessler. (*J. Gasbeleucht.*, 1912, 55, 905-906; through *Chem. Zentralbl.*, 1912, II., 1406.)—In the following modification of Houghton's method the ammonia and pyridine are first estimated together by titration with $\frac{N}{5}$ or $\frac{N}{10}$ sulphuric acid. Another sample of the gas-water is then heated, and the vapours conducted through two slightly-warmed aqueous solutions of sodium hypobromite, whereby the ammonia is oxidised to nitrogen and water, whilst the pyridine passes on and is received in $\frac{N}{10}$ sulphuric acid, the excess of which is titrated with standard alkali. The estimation may also be made by distilling the ammonia and pyridine, and adding to the distillate, after titration, sufficient sodium hydroxide solution to render the liquid slightly alkaline. It is then used for the estimation of the pyridine.

C. A. M.

Direct Determination of Rubber. C. R. Boggs. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 9, 45-58.)—The author describes a modification of Budde's method for the estimation of rubber in vulcanised or unvulcanised samples. The procedure is as follows: The sample is ground until it passes through a 20-mesh sieve; it is thoroughly extracted with acetone and the residue is dried in a reducing atmosphere, care being taken to avoid undue exposure to the air. An amount of the residue corresponding to about 0·1 grm. of pure rubber is placed in a tall shaped beaker of 300 c.c. capacity, together with 20 c.c. of toluene; the beaker is covered and the toluene boiled gently for three hours. Most of the solvent is then evaporated off on the water-bath, 50 c.c. of carbon tetrachloride are added, cooled, and then 50 c.c. of Budde's brominating mixture are introduced (*cf.* ANALYST, 1910, 35. 267). The covered beaker is allowed to remain for twenty-four hours at the ordinary temperature with frequent agitation. Fifty c.c. of absolute alcohol are then stirred in, the precipitate is allowed to settle over night, the solution is decanted through a paper filter free from chlorine, and the residue is washed first with a mixture of 2 parts of carbon tetrachloride and 1 part of absolute alcohol, and then with alcohol alone. The residue is also washed with boiling water by decantation until free from bromides, and is then transferred to the filter. It is washed with a little alcohol and allowed to dry in the air. The residue is then separated from the filter and ground to powder if necessary. The filter-paper is torn up and burned in a porcelain crucible with a little fusion mixture at a low temperature. The powdered residue is then added and mixed with about 15 grms. of the fusion mixture. The contents of the crucible are slowly brought to fusion-point, and gently fused for about ten minutes. The melt is dissolved in water, the solution filtered, acidified with nitric acid, and the bromine is estimated volumetrically with silver nitrate and thiocyanate. The amount of rubber is calculated from the bromine by the formula $\frac{C_{10}H_{16}}{Br_4}=0{\cdot}425$. No correction is applied for the sulphur in the bromide. With purified Para rubber the method gives about 93 per cent. of pure rubber; oxidised rubber is not estimated, so that the method may be applied to determine the tendency to oxidation of samples before and after exposure.

J. F. B.

Relationship between the Amount of Resins and the Viscosity of Rubber Solutions. J. G. Fol. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 9, 71-73.)—In making solutions of Castilloa plantation rubber, the author has observed that a low viscosity generally indicates a high percentage of resins, and *vice versa*. Either the resins decrease the viscosity of the rubber, or the rubber molecules themselves possess a low viscosity, when the material contains a large amount of resinous matter. The viscosities of 0·25, 0·5, and 1·0 per cent. solutions of the rubber in pure benzene are observed in the Ostwald double-bulb viscosimeter, the rate of flow of the solvent itself being taken as unity. The general validity of the above rule is shown by tables, although in certain exceptional cases other factors must come into play. After removal of the resins by boiling acetone, the viscosity of the purified rubber was in some cases higher, and in other cases lower, than that of the original sample. The heating of the rubber, without de-resination, at the same temperature and for the same time as are employed for removing the resins, caused in all cases a considerable decrease in the viscosity of the solutions. J. F. B.

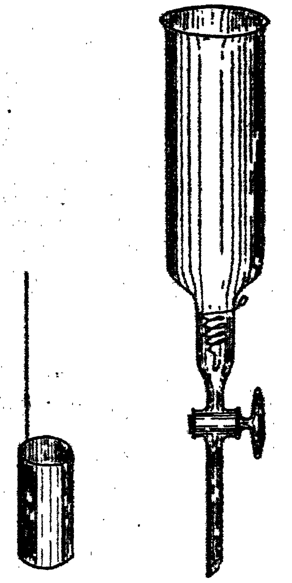
Analysis and Testing of Rubberised Fabrics. M. B. Šetlík and J. Žofka. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 9, 89-93.)—In the examination of a rubberised fabric, the cohesion, adhesion, elasticity, and resistance to rubbing and folding of the coating are first tested empirically. The cloth is then supported on a frame and water is poured on it. The degree of resistance to water is tested for several days both in the case of the original fabric and of the fabric after rubbing between the hands. The action of 5 per cent. ammonia solution and 5 per cent. acetic acid is noted as representing the behaviour of the cloth towards the liquids of the body. The weight per square metre, thickness, and tensile qualities are observed. A portion of the cloth is boiled with chloroform for three hours, and the condition of the coating is then tested by scraping. If it is swollen and comes away in large patches it contains at least 50 per cent. of caoutchouc. The separated coating is extracted with acetone, which removes resins, oils, wax, etc. The residue is saponified with alcoholic potash, which removes rubber substitutes; the remainder is caoutchouc and mineral matters. The presence of a large amount of mineral matter or rubber substitute impedes the swelling with chloroform; coatings rich in resin swell most rapidly under the action of carbon bisulphide. The fabric, freed from its coating, is washed with hot alcohol, dried and weighed, then tested for its tensile properties with the Schopper dynamometer. Fabrics with a 40 per cent. coating are quite satisfactory, even if half the coating is composed of substitutes. Zinc oxide, magnesia, and chalk are not objectionable in reasonable quantities.

J. F. B.

Expanded Meissl-Hiller Table for Invert Sugar. E. W. Rice. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 8, 47-51.)—The table is intended for use in laboratories in connection with the control of factory working where determinations are required of invert sugar in many samples of widely varying composition. As the total amount of all sugars present, as well as the ratio between cane sugar and invert sugar, affects the precipitation of copper, the calculation of results includes factors to cover the conditions in each individual analysis. These calculations are simplified by the use of these tables, which are constructed for the following conditions: Cuprous oxide is precipitated from 50 c.c. of mixed Fehling's solution by 50 c.c. of sugar solution, and the equivalent of invert sugar is calculated according to the Meissl-Hiller method. The columns of the tables contain, first, equivalent weights of copper, cuprous oxide and cupric oxide, which represent the analytical determinations made. Parallel with these are columns showing the corresponding percentages of invert sugar for quantities of 1, 2, 5, 10, and 20 grms. of the sample per 100 c.c. of the solution. These columns are each further subdivided according to the polarisation of the sample. It is essential that all the experimental conditions be maintained rigidly constant. It was found, for instance, that different beakers, apparently identical, gave variations of as much as 0·5 per cent. of the copper precipitated, owing to differing thickness and bottom area; variations between duplicate determinations, due to other causes than the beakers, were found to run about 0·36 per cent. in samples of high purity, and 0·24 per cent. in samples of low purity. J. F. B.

Electrolytic Apparatus for Use in Invert Sugar Determinations.

B. B. Ross. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 8, 75-77.)—The author describes a piece of apparatus (see Fig.) which very materially simplifies the electrolytic determination of the cuprous oxide obtained in the estimation of cupric-reducing sugars, since the electrolysis is effected without removing the oxide from the filter. The filtering funnel illustrated has a stopcock in its stem, and a spiral of platinum wire serving as the anode is fused into the lower portion of the funnel. An asbestos filter-bed is deposited in this portion of the funnel, and the cuprous oxide is precipitated and filtered by the aid of the suction-pump in the usual manner. After the oxide has been washed with hot water, the stopcock is closed and the funnel is filled about three-quarters full of dilute nitric acid, containing about 4 c.c. of acid (1·42 sp. gr.) per 100 c.c. A platinum cylinder, as shown in the illustration, is then immersed in the acid to serve as a cathode, and electrolysis is begun. A current, yielding not more than 1 c.c. of gas per minute, should be used to secure the most satisfactory deposition of the copper. Complete solution and deposition of the cuprous oxide as metallic copper are thus readily attained. J. F. B.



Inversion of Sucrose Solutions by Means of Ammonium Chloride.

F. Strohmer and O. Fallada. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 8, 85-92.)—The nitrogenous constituents which are not easily removed from impure sugar products are known to affect the accuracy of the estimation of sucrose by the

Clerget inversion method, because their optical rotation in acid liquids is different from that in neutral solution. For this reason an inverting agent which has a neutral reaction at the polarisation temperature was desirable. Ammonium chloride reacts at high temperatures as a dissociated mixture of ammonia and hydrochloric acid, and therefore inverts cane sugar. The authors' attempts to use this salt in quantitative sugar analysis have, however, led to no successful result on account of the incompleteness of the inversion under practicable conditions of working. Fifty c.c. of the normal sucrose solution, when heated for one hour at 110° C. with 30 c.c. of a solution containing 5 grms. of ammonium chloride, showed a polarisation of +3·6, as compared with an original polarisation of 49·9. The employment of zinc dust or platinum black as catalytic agents produced no more satisfactory result. Complete inversion was obtained by heating at 110° C. for forty-five minutes, 50 c.c. of a half normal sucrose solution with 30 c.c. of saturated ammonium chloride solution in presence of a trace of acid. On standing in the cold for twenty-four hours this solution gradually attained a maximum lævo-rotation exceeding that of pure invert sugar, accompanied by a coloration indicating the destruction of a certain small amount of dextrose. Further experiments seemed to indicate that the desired end might be attained by neutralising the ordinary acid inverted solution with ammonia or sodium hydroxide. J. F. B.

Detection and Differentiation of Vegetable Tannins. E. Stiasny. (*Collegium*, 1912, 483-499; through *Chem. Zentralbl.*, 1912, II., 1405-1406.)—For the detection of pyrogallol tannins in the presence of catechol tannins, 50 c.c. of the solution (as used for analysis) are boiled for thirty minutes beneath a reflux condenser with 5 c.c. of strong hydrochloric acid and 10 c.c. of 40 per cent. formaldehyde solution, and then cooled and filtered. An addition of 1 c.c. of 1 per cent. ferrous ammonium sulphate solution and 5 grms. of solid sodium acetate is now made, without shaking, to about 10 c.c. of the filtrate, and note taken whether a blue or violet coloration is produced at the bottom of the tube. Bennett's test may also be used, according to which 2 to 3 c.c. of the tannin solution are mixed with 2 to 3 c.c. of 10 per cent. sodium sulphite solution and 1 or 2 drops of 10 per cent. potassium chromate solution. All catechol tannins give a green coloration, whilst of pyrogallol tannins, myrobalans, sumach, and gallotannic acid, give a bluish-red coloration, rapidly changing to brown; and valonia, chestnut, and oak-bark extracts give a deep reddish-violet fairly stable coloration. Schell's test for the detection of mangrove extract in natural and sulphited quebracho extract consists in boiling and rapidly cooling 20 c.c. of the tannin solution, covering it with petroleum spirit and mixing it with 1 c.c. of 20 per cent. cobalt chloride solution and 1 c.c. of strong ammonia solution. Quebracho extracts give a grey-green, mangrove extracts a brown-violet, mimosa a grey-blue, and algarobilla a light brown, coloration in this test. It is not always possible, however, to detect 10 to 20 per cent. of mangrove extract with certainty in this way. In Hoppenstedt's test 25 c.c. of the tannin solution are gradually mixed with 25 c.c. of a 1 per cent. quinine hydrochloride solution and filtered, and 5 c.c. of the clear filtrate of non-tannin constituents are treated successively with 1 c.c. of concentrated acetic acid, 2 c.c. of acetone, and 5 c.c. of ethyl acetate, and shaken. In the presence of mangrove extract the lower layer will be dark yellowish-brown, but colourless in the case of other tannins. In the author's experience certain quebracho extracts and wood pulps may also yield a faint yellow coloration in the lower layer even in the absence of mangrove extract. C. A. M.

Tests to determine the Commercial Value of Wood Preservatives H. F. Weiss. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 13, 279-300.)—From 40 to 90 per cent. of the total cost of treating wood is in general due to the preservative; and as in ordinary practice it is usual to inject about 10 pounds of the oil per cubic foot of wood, although about $\frac{1}{2}$ pound will prevent fungoid growth, it is obvious there is a wide margin for safe reduction. The present paper exhaustively summarises the conclusions arrived at as a result of an experimental investigation of the following properties: (1) The important chemical and physical properties of the preservative; (2) the effect of the preservative on the strength of the wood; (3) the ability of the preservative to penetrate and diffuse through the wood; (4) the permanency of the preservative after its injection, including a study of its volatility and capability of being leached; (5) the combustibility of the treated wood; (6) the toxic efficiency of the preservative in inhibiting the growth of wood-destroying fungi; (7) the corrosive action of the preservative on steel; (8) the effect of the preservative on paint applied to the wood subsequent to treatment.

A description is given of the methods by which each of the above points was investigated, accompanied by illustrations of the special apparatus and plant required for many of them. For the results which follow, a greater accuracy than plus or minus 10 per cent. cannot be claimed, chiefly because of the variables inherent in wood. The results themselves are too numerous for adequate abstraction, but a few of the general conclusions follow. The depth to which oils can be impregnated varies inversely as some function of their viscosity. To secure the best results, the wood and the preservative should be heated to the optimum temperature during the pressure period. With other than water-soluble preservatives the treatment should not be made too rapidly. With coal-tar creosote the fractions of greatest stability are the least toxic. Present practice favours the retention in treated wood of the more volatile fractions by an admixture of the more stable ones. It would appear that in practice there is being forced into wood about two and a half times as much zinc chloride, and fifty times as much coal-tar creosote, as is necessary to prevent decay; more thorough diffusion of less preservative rather than saturation of the outer layers of the wood is thus required. A prolonged seasoning of treated wood considerably raises its ignition temperature. Wood treated with the water-soluble salts mentioned in the tests, although less difficult to ignite, yet proved to be slower burning and more readily extinguishable than untreated wood. All the metallic salts used were much more corrosive in their action on steel than coal-tar creosote. All the oils tested rendered the wood unfit for subsequent painting, copperised oil being the least objectionable. H. F. E. H.

INORGANIC ANALYSIS.

Estimation of Arsenic in Organic Compounds. T. St. Warunis. (*Chem. Zeit.*, 1912, **36**, 1205.)—For the analysis of organic arsenic compounds the combustion of which is difficult, from 0·2 to 0·4 grm. of the substance is stirred, with a platinum wire, into an intimate mixture of 10 grms. of potassium nitrate and 5 grms. of sodium peroxide in a large nickel crucible, covered with a layer of the mixture, and heated gradually to fusion. When quite fluid the melt is stirred, maintained for a short time in a state of fusion, and dissolved in hot water. The solution, which contains all the arsenic as arsenate, is acidulated with hydrochloric acid, filtered if necessary, and neutralised with ammonia. The arsenic is determined as $Mg_2As_2O_7$ by means of magnesia mixture. It may also be estimated volumetrically as uranium arsenate (Brügelmann, *Zeit. anal. Chem.*, 1877, **16**, 20) in the dissolved melt. The acidulated liquid is first rendered alkaline to litmus-paper with sodium hydroxide or ammonia, and then strongly acid with acetic acid, and made up to 100 c.c.; not more than 50 c.c. of this is taken for titration with a uranium solution containing about 20 grms. uranium oxide per litre. Non-volatile compounds which are readily decomposed are fused with potassium nitrate and sodium peroxide in a hard glass tube, 35 cm. long, closed at one end. This is charged, in succession, with a 2-cm. layer of sodium peroxide, the mixture containing the substance, 3 cm. of sodium peroxide, a plug of asbestos, and anhydrous sodium carbonate for the rest of the tube. The sodium carbonate is cautiously heated first, then the sodium peroxide on each side of the substance, and finally the whole tube, the cooled contents of which are dissolved in water and treated as above.

O. E. M.

Determination of Small Quantities of Arsenic by the Gutzeit Test. W. S. Allen and R. M. Palmer. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 9-17.)— Under standard conditions the Gutzeit test gives results as accurate as those obtainable by the Marsh-Berzelius method, which requires much more of the analyst's time. The influence of small amounts of iron is recognised and met by the addition of from 0·05 to 0·1 grm. of iron as ferric salt in every test. The ferric salt is reduced by stannous chloride, and the presence of both iron and tin insures an even and adequate evolution of hydrogen, and longer stains are obtained in this way than when omitting the addition of iron.

The apparatus used by the authors consists of a 60-c.c. bottle, the rubber stopper of which has a single hole. Through this hole passes the tapered end of a tube 7 cm. long and 1·2 cm. in diameter. This tube holds a strip of dry lead acetate paper, and its rubber stopper carries a similar but shorter (4 cm.) tube, which contains glass-wool moistened with a solution of lead acetate. Through the rubber stopper of this upper tube passes a tube 10 cm. long, 4 mm. in internal diameter, and constricted in the middle to support in its upper half the strip of mercuric chloride paper. This is made by dipping filter-paper in 0·5 per cent. mercuric chloride solution, removing excess of solution by use of a rubber roller, and drying rapidly on a cloth in an oven at 100° C. The edges of the paper are trimmed off and rejected, and the rest is cut into strips about 7 cm. long and as nearly as possible 4 mm. wide.

The reagents required are (1) zinc in pieces which pass a screen of three holes to the linear inch, but not one of six holes to the inch. This zinc is washed in hydrochloric acid (1 : 1) and preserved under water. (2) Sulphuric acid (1 : 4), containing 100 grms. of sodium chloride in each litre, the reduction of iron proceeding more readily in presence of hydrochloric acid. (3) Stannous chloride solution, made by dissolving 80 grms. of the crystallised salt in 100 c.c. of water and 5 c.c. of hydrochloric acid. (4) A solution of iron alum.

The solution to be analysed, which must not contain more than traces of sulphide or sulphite, nor nitric acid, chlorine, etc., has iron alum solution added to it in amount corresponding to about 0·05 to 0·1 grm. of ferric oxide. This is followed by 0·5 c.c. of stannous chloride solution, and the mixture is boiled until the iron is all reduced to the ferrous state, when it is cooled and transferred to the Gutzeit apparatus, together with fifteen pieces of zinc and 10 c.c. of the sulphuric acid mixture. After gently shaking its contents, the bottle is placed in a water-bath at a temperature within a few degrees of 75° C., and after thirty minutes the sensitised paper is withdrawn, dipped in melted paraffin, and compared with a series of standards ranging from 0·001 to 0·02 mgrm. Standards preserved in sealed tubes over phosphoric anhydride, and kept in the dark, show no perceptible change in several months. G. C. J.

Titration of Arsenic Acid. A. W. C. Menzies and P. D. Potter. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 367-371.)—When arsenic acid is titrated with sodium hydroxide, using phenolphthaleïn as indicator, a rather uncertain end point is reached when about two-thirds of the hydrogen has been replaced by sodium. Quite sharp end points, corresponding to the formation of disodium arsenate, may be secured as follows. To the solution to be titrated 15 c.c. of a cold saturated solution of barium chloride is added, and the mixture is boiled to expel carbon dioxide. It is then titrated with $\frac{N}{1}$ caustic alkali, with stirring after each addition, until the locally formed precipitate becomes slow in redissolving. The walls of the vessel are now, if necessary, scratched below the surface of the clear solution until the liquid on stirring appears lustrous with fine crystals, after which the titration is completed. Under the above conditions the results are as accurate as the results of a volumetric analysis can be, but the scratching action of a stirring rod to induce the formation of a precipitate of dibarium arsenate is necessary, or the proper end-point may be much overstepped. In fact, with solutions of known titer, it is possible to run in alkali until practically the whole of the arsenic exists as triarsenate before the solution assumes a pink tint. G. C. J.

Volatility of Arsenious Chloride. J. I. D. Hinds. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 227-231.)—Experiments are described which show that the quantity of arsenious chloride lost on boiling its hydrochloric acid solution is a function of the concentration of the arsenic and the acid, and that the rate of loss is much smaller than is supposed by some authors who issue warnings against boiling acid arsenious solutions even in qualitative work. For example, a $\frac{N}{10}$ solution of arsenious chloride, $\frac{N}{1}$ as regards acidity, may be concentrated to half-bulk without loss of more than one-thousandth part of the arsenic present. G. C. J.

Detection of Traces of Arsenic and Antimony. D. R. Staddon. (*Chem. News*, 1912, **106**, 199.)—From 0·5 to 2 grms. of the substance to be examined are dissolved in about 5 c.c. of water. Acids and acid salts are first neutralised. An equal weight of sodium hydrosulphite ("blankit") is added to the solution and the liquid warmed. Rapid reduction to metallic arsenic or antimony, as the case may be, occurs, the precipitate varying from a light to dark brown colour according to the quantity of substance present. The precipitate of arsenic is dissolved by sodium hypochlorite, while that of antimony is insoluble and remains suspended. One part of sodium arsenite in 50,000 parts of water may readily be detected by this method.

A. R. T.

Gravimetric Estimation of Beryllium. B. Bleyer and K. Boshart. (*Zeitsch. anal. Chem.*, 1912, 51, 748-755.)—The precipitation of beryllium as hydroxide is best effected in the cold, using but a small excess of ammonia, and taking care to have ammonium chloride present. Large amounts of the latter introduce no error, but 1 grm. is usually more than sufficient. The precipitate is washed with a hot dilute solution of ammonium nitrate containing a few drops of ammonia, and any precipitate which adheres to the precipitating vessel is removed with a fragment of ash-free paper. The filter-paper and precipitate are transferred to a platinum crucible without previous drying, and when the paper is burnt the crucible is covered and heated in the blowpipe flame for ten minutes. The precipitation should take place in a vessel of porcelain, and not in a glass beaker. Platinum serves as well as, but no better than, porcelain. Precipitation in hot solution, followed by boiling to eliminate excess of ammonia, as is often recommended, may lead to beryllium being underestimated by as much as 20 per cent. Precipitation by means of ammonium sulphide (*cf.* Crookes' "Select Methods," 1894, 150) instead of ammonia gives equally good results, but only if the precipitation be made in the cold and a considerable time allowed to elapse before filtration. Glassmann's method (*Ber.*, 1906, 39, 3368), depending on the use of a mixture of potassium iodide and iodate, gives accurate results, but occupies more time than the ammonia method. G. C. J.

Sensitive Reaction of Free Bromine. G. Denigès. (*Compt. rend.*, 1912, **155**, 721-723).—An aqueous solution of rosaniline bisulphite gives a purple or violet coloration, and eventually a violet precipitate with free bromine. The reagent is prepared by adding 10 c.c. of sodium bisulphite solution (sp. gr. 1·29) to 1 c.c. of a 0·1 per cent. aqueous solution of fuchsin, followed, after about five minutes, by 20 c.c. of hydrochloric acid (sp. gr. 1·18). The compound of rosaniline and bromine is insoluble in water, ether, benzene, and petroleum spirit; sparingly soluble in rosaniline bisulphite solution, but very soluble in methyl, ethyl and amyl alcohols, acetone, acetic acid, and carbon tetrachloride. The solutions of the compound show a characteristic spectrum with two absorption bands, one in the blue and the other in the orange part of the spectrum. The action of bromine in the bisulphite is inhibited by adding hydrogen peroxide. In applying the test, from one drop to several c.c. of the solution under examination are shaken with a mixture of 2 c.c. of the reagent, 2 c.c. of hydrogen peroxide solution, and 1 c.c. of chloroform, and in the presence of as little as as 0·01 mgrm. of bromine a violet tint will be produced. Atmospheric air is tested by being made to bubble through the mixture, or test-papers prepared by saturating filter-paper with the reagent and drying it in the air, may be used. Before use the paper is slightly moistened with water containing 1 to 2 per cent. of hydrochloric acid. The test may be used in the presence of a large excess of chlorides or iodides, and may be made quantitative by colorimetric comparison with standard solutions of bromine. The presence of combined bromine may be detected, *e.g.*, in 2 c.c. of a solution of potassium bromide, corresponding to 0·013 mgrm. of bromine, by adding 0·5 c.c. of a 10 per cent. solution of potassium chromate and 0·5 c.c. of sulphuric acid, shaking the mixture with a fragment of marble to cause a slow evolution of gas, and suspending a moistened and acidulated test-paper 2 to 3 cm. above the surface of the liquid. C. A. M.

Estimation of Free Carbon Dioxide in Water by Titration with Alkalis in the Presence of Phenolphthaleïn. J. Tillmans and O. Heublein. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 429-449.)—The authors show by their experiments that free carbon dioxide in water can be titrated accurately by dilute alkali solutions, using phenolphthaleïn as indicator. The quantity of carbon dioxide present in the water should not exceed 100 mgrms. per litre, and the temporary hardness must not exceed 10 (German) degrees; otherwise the water must be diluted with neutralised distilled water previous to the titration. The quantity of phenolphthaleïn added is also of importance, and it is recommended that 1 c.c. of a 0·035 per cent. alcoholic solution of the indicator be used for every 200 c.c. of water. The titration must be carried out in a closed flask (*cf.* ANALYST, 1911, **36**, 362).

W. P. S.

Use of Higher Phenols in Testing for Free Lime in Portland Cement. D. C. McFarland and H. F. Hadley. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 5, 83-90.)—Experiments described in detail have shown that the salts formed by calcium with phenol and homologous compounds are only sparingly soluble in any of the ordinary solvents. The speed with which different phenols react with free lime shows great variations. For example, combination takes place immediately in the case of *p*-cresol, whilst *β*-naphthol and phenol do not react so rapidly, and there is only a slight reaction with *α*-naphthol, *o*-ethyl phenol, carvacrol, 1-3-4 xylenol, *o*-cresol, and *p*-nitro-cresol. Of these phenols, only *β*-naphthol and *p*-cresol appeared to react to the same extent as phenol. A rough estimation of the free lime in cement (when small in quantity) may be made by shaking 3 grms. of the sample for an hour with a mixture of 21 c.c. of absolute alcohol, 30 drops of *p*-cresol, and 0·5 c.c. of water, then filtering the liquid, washing the residue with water until colourless, evaporating the filtrate and washings, igniting the residue, and weighing it as calcium oxide. If much free lime is present, however, the results thus obtained are too low, and do not agree well in duplicate estimations. A mixture of 1 c.c. of *p*-cresol, 3 c.c. of absolute alcohol, and 4 drops of water, gives a reagent which reacts much more rapidly than White's phenol reagent (Analyst, 1909, **34**, 71) for the detection of free lime. For example, an abundance of pale red crystals easily observed without polarised light, was obtained in two to three minutes from cements containing 5 per cent. of free lime, and by means of this test it was proved that in old cements free lime may still be present, its particles having been protected by an outside coating formed by the action of the air. For practical purposes this modification of the test is regarded as almost too delicate. C. A. M.

Estimation of Chlorous Acid (Chlorites). G. Lasègue. (*Bull. Soc. Chim.*, 1912, **11**, 884-886.)—The process is based on the insolubility of lead chlorite in alcohol; this salt is slightly soluble in water, but practically insoluble in 80 per cent. alcohol. It slowly oxidises alcohol at the ordinary temperature, but this action is very slight within a period of one hour. The chlorite is dissolved in water, an excess of lead nitrate solution is added, and the mixture is stirred. The mixture then receives the addition of six times its volume of 85 per cent. alcohol, and, after the lapse of one hour, the precipitate is collected on a weighed filter, dried over sulphuric acid under reduced pressure, and weighed. When the chlorite solution contains an excess of alkali, as is often the case when the chlorite has been prepared by passing the gas into an alkali solution, magnesium nitrate is added before the estimation is carried out. The presence of small quantities of chlorides does not interfere with the estimation, and chlorites, chlorides, hypochlorides, and chlorates, may be estimated in a mixture containing the same in the following way: In an aliquot portion of the solution the chlorite is estimated as described. Another portion of the solution is treated with potassium iodide and hydrochloric acid, the iodine liberated being a measure of the chlorite plus hypochlorite. In order to prevent the action of the chlorate on the iodide, the solution must be very dilute, and contain not more than 0·3 grm. of the salt per 500 c.c. In a third portion of the solution the chloride, resulting from the reduction of chlorite and hypochlorite by a standardised alkali arsenite solution, together with the chloride originally present, is titrated with silver nitrate solution. After removing the silver chloride by filtration, the chlorate in the filtrate is reduced by means of sodium nitrite, and the resulting chloride also titrated. The following results were obtained with a mixture of the four salts, the figures *in parentheses* showing the actual quantities present: Chlorine, as chloride, 13·1 (12·5); as hypochlorite, 11·92 (12·5); as chlorate, 37·48 (37·5); as chlorite, 37·34 (37·5). W. P. S.

Quantitative Separation and Estimation of Copper by Means of Hydroxylamine Hydrochloride. A. Bayer. (*Zeitsch. anal. Chem.*, 1912, **51**, 729-735.)—To the solution of copper as sulphate, which may conveniently contain about 0·3 grm. copper in 50 c.c., there is added about 50 c.c. of 25 per cent. solution of Rochelle salt, 25 c.c. of a 15 per cent. solution of sodium hydroxide, and 2 c.c. of a 5 per cent. solution of hydroxylamine hydrochloride. The mixture is boiled for a minute, and the cuprous oxide is filtered off in a tared Soxhlet tube, ignited to cupric oxide and weighed. Neither antimony, zinc, bismuth, lead, iron, arsenic, nor tin interferes, and quantities of ammonia or of nitrates corresponding to 1 grm. of ammonium chloride or sodium nitrate do not influence the results, which are exact. Mercury, if present, is partly precipitated, but introduces no error if the cuprous oxide is ignited to cupric oxide. Silver, gold, platinum, manganese, cobalt, and nickel should be absent. If the three latter metals, but not gold, platinum, or silver, be present, they are easily eliminated by precipitating the copper first as sulphide in acid solution. The precipitate of copper and other sulphides, together with the filter-paper, is oxidised by nitric acid, and the solution taken down to fuming with sulphuric acid, then diluted, and copper precipitated as described. G. C. J.

Electro-analysis of Copper, Antimony, Bismuth, and Tin with Acidified Chloride Electrolytes. E. P. Schoch and D. J. Brown. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 21, 81-91.)—Copper, tin, antimony, and bismuth can be deposited quantitatively and fairly quickly from hydrochloric acid solutions, provided a reducing agent, such as hydroxylamine hydrochloride, is present. An alloy of copper and tin may be analysed as follows: The alloy is dissolved in *aqua regia*, and the solution evaporated to expel free chlorine and oxides of nitrogen. Hydroxylamine hydrochloride (2 grms.) and about 10 c.c. of concentrated hydrochloric acid are then added, and the solution is diluted to 200 c.c. and electrolysed, using the Sand-Fischer apparatus (*Zeitsch. Electrochem.*, 1907, **13**, 469), and limiting the cathode potential to −0·4 volt (against a normal calomel electrode). The copper is deposited in from twenty to forty minutes, and the end-point is recognised by the fact that the current must be reduced to zero to prevent the cathode potential exceeding the limiting value. The beaker is lowered, and the cathode is washed, detached, dipped in alcohol and ether, dried above a flame, and weighed. It is then replaced, 3 grms. of hydroxylamine hydrochloride is added to the electrolyte, and the tin is deposited by means of a constant current of 1·5 amps., the end point, which is reached in twenty to forty minutes, being ascertained by means of hydrogen sulphide. With more than 0·7 grm. tin present in 200 c.c., a lower current density is necessary to ensure good deposits.

In the electrodeposition of antimony, the evolution of hydrogen and attendant stibine must be avoided, the temperature must be above 50° C. or "explosive" antimony may be deposited, and unnecessarily high temperatures must be avoided, as they increase the tendency of antimony solutions to hydrolyse. Good results are obtained at 50° to 75° C. with 20 to 25 c.c. of hydrochloric acid and 2 grms. hydroxylamine hydrochloride in each 200 c.c. of electrolyte, and a cathode potential of −0·4 volt (against a normal calomel electrode); 0·5 grm. may be deposited in ten minutes with an error not exceeding 1 mgrm.

To obtain a good deposit of bismuth it is necessary to limit the cathode potential to −0·25 volt until most of the metal has been deposited, and then to allow it to rise gradually to −0·6 volt during the last few minutes. At 55° to 75° C., with 5 c.c. hydrochloric acid and 2 grms. hydroxylamine hydrochloride present in 200 c.c. of electrolyte, 0·5 grm. of bismuth may be deposited in twelve minutes with an error not exceeding 0·8 mgrm. G. C. J.

Influence of Lead on the Ferrocyanide Titration of Zinc. V. Lenher and C. C. Meloche. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 279-284.)—Contrary to the statements of several authors (*e.g.*, Seaman, *J. Amer. Chem. Soc.*, 1909, **29**, 207), lead does not interfere with the estimation of zinc by ferrocyanide as that operation is usually carried out—namely, in presence of hydrochloric acid and ammonium chloride. The concentration of the latter may be anything between 1 and 20 grms. in 200 c.c., but concentrations as high as 20 per cent. give rise to an indistinct end-point. Indistinct end-points also result from too low or too high concentration of acid, but sharp end-points are obtained over a fairly wide range—namely, from 1·5 to 6 per cent. free hydrochloric acid. Ten c.c. of concentrated hydrochloric acid and 10 grms. of ammonium chloride in 200 c.c. are convenient quantities to have present. The best indicator for general use is a 5 per cent. solution of uranium nitrate. A 1 per cent. solution of ammonium molybdate is more sensitive, but its application is limited.
G. C. J.

Rapid Determination of Magnesia in Limestone by Means of the Hydrogen Electrode. J. H. Hildebrand and H. S. Harned. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 217-225.)—The method depends on the fact that, when caustic alkali is added to a neutral solution containing salts of calcium and magnesium, the concentration of hydroxyl ions increases very slowly until all the magnesium is precipitated as hydroxide, and then increases rapidly to the point at which calcium hydroxide begins to separate. The method therefore consists essentially of the titration of the magnesium, making use of the above-described sharp rise in hydroxyl ion concentration to determine the end-point. In practice it is more convenient to measure the diminishing hydrion concentration which is a reciprocal function of the hydroxyl ion concentration. The necessary apparatus includes an easily constructed form of hydrogen electrode, a calomel electrode, a voltmeter reading from 0·01 to 1 volt, any fairly constant cell with E.M.F. not less than 1 volt, a sliding rheostat or bridge wire of fairly high resistance, and a galvanometer or Lipmann electrometer as zero indicator. The limestone is dissolved in hydrochloric acid, and the solution is freed from carbon dioxide and titrated with normal caustic alkali (free from carbonate) in a beaker into which dip the hydrogen electrode and the syphon from the calomel electrode. By the use of methyl-orange, the neutralisation of the free acid may be quickly effected. From this point onwards electrometric readings are taken and the observed E.M.F. in centi-volts plotted as ordinates against the burette readings as abscissæ. With a small addition of alkali beyond that necessary to change the colour of methyl-orange, the E.M.F. rises rapidly, and a further small addition causes the curve to take a nearly horizontal direction. Larger additions of alkali may now be made between each reading until the curve assumes a steeper gradient, when readings are taken at every 0·2 c.c. When the gradient of the curve begins to diminish instead of increasing, the titration is at an end. The reading of the burette at this moment, less the reading when curve changed its nearly vertical direction to a horizontal one, is the measure of the magnesium present. The method is accurate to within 2 per cent. of the magnesium present. The paper includes a diagram of the whole apparatus, a drawing of the hydrogen electrode, and a typical curve.
G. C. J.

New Volumetric Method for the Estimation of Nitrites, and for the Separation of Nitrous Acid from Nitric Acid. W. M. Fischer and N. Steinbach. (*Zeitsch. anorg. Chem.*, 1912, 78, 134-140.)—The method depends on the readiness with which nitrous acid is converted into its methyl ester (b.-pt. −12° C.). It is sufficient to add a nitrite solution drop by drop to a mixture of dilute sulphuric acid, methyl alcohol, and water, through which a current of air is bubbled, in order to convert the whole of the nitrite into the ester and remove the latter as it is formed. If a known quantity of sulphuric or hydrochloric acid is present in the mixture, the difference between this and the amount remaining at the end of the experiment is a measure of the nitrous acid.

None of the nitrite is oxidised to nitrate during the reaction, and the mixture does not yield a reaction for nitrate at the end of the operation, unless nitrates were present originally in the nitrite solution. The estimation is carried out most conveniently by placing the nitrite solution and 45 c.c. of methyl alcohol in a 300 c.c. flask. The flask is closed with a rubber stopper, through which pass the stem of a tapped funnel, an inlet tube reaching to the bottom of the flask, and a delivery tube, the latter being attached to an exhaust pump. A measured excess of $\frac{N}{10}$ hydrochloric or sulphuric acid and 1 c.c. of methyl alcohol are placed in the funnel, and allowed to drop into the flask, while air is drawn through the apparatus. When all the acid has been introduced, the current of air is passed through the flask for a further five minutes, and the excess of acid is then titrated with $\frac{N}{10}$ sodium hydroxide solution (free from carbonate), using phenolphthaleïn as indicator. If, instead of a nitrite solution, a mixture of nitrite and nitrate is under examination, the nitrate may be estimated subsequently by adding to the neutralised contents of the flask 3 grms. of Devarda's alloy and 20 grms. of potassium hydroxide, and estimating the ammonia formed in the usual way. The method may also be employed for estimating nitrites and carbonates together, as the latter will neutralise an equivalent quantity of the acid in the flask; a separate estimation of the nitrous acid, for instance, by titration with permanganate, will allow the amount of carbonate to be calculated. Results of analyses are given, which show that the method is accurate. W. P. S.

Determination of Oxygen in Iron and Steel by Reduction in an Electric Vacuum Furnace. W. H. Walker and W. A. Patrick. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 21, 139-148.)—Ledebur's method for the determination of oxygen in iron and steel gives low results because hydrogen at a red heat fails to reduce alumina and silica, and the reduction of oxides of manganese is far from complete. The method now described depends on the reduction of all the oxides to carbides by heating them with excess of carbon to a high temperature in an electric furnace, the oxygen being given off quantitatively as carbon monoxide. The furnace illustrated in the paper is a vacuum furnace of the Arsem type (made by the General Electric Company), and requires 14 kw. (200 ampères at 70 volts) to operate it. The charge of metal taken for the test is 20 to 25 grms., and is contained in a graphite crucible and covered with about 5 grms. of powdered graphite. The furnace is exhausted to 0·01 mm. by means of a rotary oil-pump working in series with a Geryk pump, the temperature being raised to 500° to 600° C.

towards the end of the operation. The furnace is allowed to cool, and nitrogen dried by means of sulphuric acid and phosphoric anhydride, is admitted, until the gas pressure is about half an atmosphere. The nitrogen is then exhausted by the pump, heating to 500° to 600° C., when the pressure is reduced to 0·01 mm. The charge is now fused, an operation which requires three or four minutes. A mica window enables the operator to watch the fusion and switch off the current if, as is often the case, there is a tendency to violent ebullition at the moment of complete fusion. After allowing the charge to cool slightly, the circuit is completed once more, and heating continued for twenty minutes. A portion of the gaseous contents of the furnace is now transferred to a glass container by means of a Töpler pump, and is dried and passed over iodine pentoxide, heated to 130° C., the liberated iodine being retained by a solution of potassium iodide and titrated with thiosulphate. The fraction of the gaseous contents of the furnace taken for the final test is determined by the readings of a differential gauge attached to the furnace. When operating as described, the blank to be subtracted from the oxygen found lies between 0·012 and 0·013 grm. This is mainly due to traces of oxygen, which obstinately resist removal during the preliminary exhaustion. One steel examined showed 0·10 per cent. oxygen by the new method and nil by Ledebur's; another 0·23 by the new method and 0·069 by Ledebur's. G. C. J.

Determination of Ozone by Means of Cadmium Potassium Iodide. C. Baskerville and W. J. Crozier. (*J. Amer. Chem. Soc.*, 1912, **34**, 1332-1337.)—Schönbein's method for the estimation of ozone by determining the iodine liberated from metallic iodides is quite generally employed, although open to objections; and the fact that when acid potassium iodide solution is used the reaction is quite unreliable is well known. The authors have confirmed and amplified the work of Baskerville and Hamor, and found that pure cadmium potassium iodide ($CdKI_3.H_2O$) is much more stable than potassium iodide, and is a satisfactory reagent for the estimation of ozone where neutral solutions of potassium iodide cannot be employed.

The cadmium potassium iodide of the above composition must be pure, free from cadmium iodide, potassium iodide, and excess of water. Such a reagent was found to be as delicate as potassium iodide solution of the same strength for hydrogen peroxide and ozone. In the estimations the air to be ozonised was passed from a gasometer through the ozoniser and a bulb (Meyer's) apparatus for holding the solution of the reagent, this apparatus being connected by a ground-glass joint. Similar results were obtained with neutral and acid solutions of $\frac{N}{10}$ strength of the cadmium reagent and neutral potassium iodide, while the results with acid potassium iodide were higher. Satisfactory results were obtained by absorbing the ozone in turpentine oil, liberating the iodine by agitating the oil with the iodide solution, and titrating the iodine with $\frac{N}{10}$ sodium thiosulphate solution. The results with alkaline iodide solutions were unsatisfactory. Cadmium potassium iodide solution, acidified with hydrochloric acid, is more reliable than potassium iodide in neutral solution, but its employment is only necessary when the ozone is absorbed in turpentine oil. A. R. T.

Potash Analysis (Potassium-Sodium Cobaltinitrite Method). E. A. Mitscherlich and H. Fischer. (*Landw. Vers.-Stat.*, 1912, **78**, 75-86; through *Chem. Zentralbl.*, 1912, II., 1312.)—The authors have made further investigations on the composition of the precipitate of potassium sodium cobaltinitrite, and on the potassium factor deduced therefrom (see also *Landw. Vers.-Stat.*, 1910, **76**, 139). The composition of the precipitate changes with the concentration of the reagent. With increasing proportions of the reagent (0·3 grm. of cobalt chloride + 0·5 grm. of sodium nitrite) the quantity of sodium entering the molecule of the product increases at the expense of the potassium. The composition of the salt approaches a limiting value asymptotically after a certain excess of reagent has been added. If this excess amounts to thirty or more times as much sodium as potassium, the variations in composition of the precipitate fall within the limits of error of the estimation. The method, therefore, gives satisfactory results provided a large excess of precipitating reagent be taken; if more than 30 parts of sodium to 1 of potassium be used, it is immaterial how great the excess may be. The reduction factor to be used for calculating from $\frac{N}{10}$ potassium permanganate to potash (K_2O) is 0·000161, not 0·000157 as previously given (*loc. cit.*).

The authors have also shown that their method is applicable for the estimation of potash in fertilisers. With quantities of potash from 0·002 grm. of K_2O upwards, the probable error is, on the average, less than ± 1 per cent. of the quantity to be estimated. J. F. B.

Estimation of Potassium in Potassium Silicate. E. Wilke-Dörfurt. (*Zeitsch. anal. Chem.*, 1912, **51**, 755-760.)—For the estimation of potassium in "phonolith," a low grade potash manure in which the potash exists as silicate, Verweij (*Zeitsch. anal. Chem.*, 1909, **48**, 760) recommended fusion with calcium carbonate and ammonium chloride as in the ordinary Lawrence Smith method for the estimation of alkalis in silicates, followed by the direct precipitation of the potassium in the aqueous extract from the fusion, without previous separation of lime. The convenience of the Lawrence Smith method, which gives the alkalis as chlorides, is admitted, as well as its accuracy, but analytical numbers are now brought forward to show that it is necessary to remove from the solution the very large amounts of calcium chloride which result from the reaction in the crucible, before proceeding to precipitate potassium as platinichloride. If calcium salts are not removed 9 per cent. of potash may be reported when only 7 per cent. is present. Such differences have occurred between the results of different German chemists reporting on one and the same sample of "phonolith." Fresenius and Brinton showed (ANALYST, 1911, **36**, 178) that potassium could be estimated with fair accuracy as platinichloride in presence of an equal weight of calcium, but in the solution resulting from the fusion of phonolith with calcium carbonate and ammonium chloride the ratio of calcium to potassium is much greater than 1 : 1. G. C. J.

Mechanical Analysis of Arid Soils. W. Beam. (*Cairo Sci. J.*, 1912, **5**, 107-119; through *Bull. of the Bureau of Agric. Intell. and of Plant Diseases*, 1912, **3**, 1721.)—Soils of tropical and arid regions differ from those of other districts in their calcium carbonate content, and especially in the amount of calcium sulphate in the subsoil and in the low humus content which in semi-arid regions may reach less than 0·5 per cent. Clay soils—*i.e.*, those which contain about 50 per cent. of clay—yield only about half the clay in the form of a suspension when shaken in a rotating machine (100 motions per minute), unless a 2 per cent. solution of sodium carbonate is employed in place of water. A threefold division is recommended for grading soils of this type: (*a*) particles of more than 2 mm.; (*b*) particles lying between 2 mm. and 0·002 to be separated into particles of 0·2-2 mm., 0·02-0·2 mm., and 0·002-0·02 mm.; (*c*) particles of less than 0·002 mm., the clay. The following procedure is adopted: 100 grms. of air-dried soil are passed through a sieve with 2 mm. round holes. The fine soil is afterwards separated by a 1 mm. sieve and the sandy residue then washed. The fine portion is dried at 110° C., 5 grms. are weighed out, treated with a 2 per cent. solution of sodium carbonate, mixed by means of a cut-down camel-hair brush and transferred to a cylinder, the volume being made up to 10 cm. The whole is allowed to stand for eight to twelve hours, and the supernatant liquor is decanted off at intervals of eight hours, being replaced by 0·5 grm. sodium carbonate, and more water. After three decantations the residuum is again mixed up by means of the brush, and after the removal of the clay, is washed with distilled water in a 200 c.c. vessel, and again left to settle for eight hours. The residue is then dried and weighed, the amount of clay being determined from the difference in weight, the soluble salts and humus being deducted if necessary. H. F. E. H.

Modification of the Method of Mechanical Soil Analysis. C. C. Fletcher and H. Bryan. (*U.S. Dep. Agric. Bureau of Soils Bull.*, 1912, No. 48; through *Bull. of the Bureau of Agric. Intell. and Plant Diseases*, 1912, **3**, 1720.)—Two thousand soils yearly are analysed and added to the collection of 20,000 samples in the Bureau, most of which have been submitted to mechanical analysis. The following are the limits of mechanical grades: Fine gravel, 2-1 mm.; coarse sand, 1-0·5 mm.: medium sand, 0·5-0·25 mm.; fine sand, 0·25-0·1 mm.; very fine sand, 0·1-0·05 mm.; silt, 0·05-0·005 mm.; clay, 0·005-0·0009 mm. The modified method is as follows: The field samples are passed through a 2 mm. sieve, and the fine earth is then dried in aluminium dishes for two hours in an electric oven, cooled in a desiccator, and 5 grm. samples weighed out into 8-ounce bottles. To each bottle is added 2 ounces of water and a few c.c. of ammonia. All are then shaken in a shaking machine for at least seven hours. Each sample is then brought into suspension by the use of a compressed water-jet, and the sands allowed to settle, the time required being controlled by microscopic examination. The liquid portion is decanted into a tube and centrifuged until no silt particles remain in suspension. The clay suspension is poured into a porcelain vessel and the treatment repeated. Thus, finally, the sands are left in the bottles, the silt in the centrifuge, and the clay in the porcelain vessels. The sands are transferred to platinum dishes, dried, and their total weight obtained, after which they are separated by three minutes' shaking in suitably meshed sieves. The silts are then transferred to platinum dishes, dried, and weighed, and the clay water evaporated in enamelled-ware cups of 300 c.c. capacity, the clay being weighed in them without further transference. The clay may also be determined by difference with an accuracy of 1 per cent. Detailed descriptions and drawings of all the apparatus are given. H. F. E. H.

Action of Boiling Sulphuric Acid on Platinum. Le R. W. McCay. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 351-359.)—Boiling sulphuric acid dissolves platinum in considerable amounts. It is shown that the oxygen of the air is not concerned in this action. In prolonged evaporations of sulphuric acid solutions in platinum basins, the action of the acid on the platinum is almost entirely prevented by the presence of a globule of sulphur, and it is suggested that any possible loss of platinum in the parting of silver-platinum alloys with boiling concentrated sulphuric acid could be avoided by a similar expedient. G. C. J.

Note by Abstractor.—That platinum is lost in the ordinary platinum assay has been shown by Steinmann (ANALYST, 1911, **36**, 605).

New Colorimetric Method for Titanium. V. Lenher and W. G. Crawford. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 285-293).—For the estimation of very small quantities of titanium, a method depending on the use of a solution of thymol in sulphuric acid is preferable to the hydrogen peroxide method. The depth of colour produced, which is strictly proportional to the amount of titanium present, is twenty-five times as intense as the colour produced by hydrogen peroxide. The substance to be tested is fused with potassium bisulphate, and the fusion is taken up in concentrated sulphuric acid, cooled, and treated with the reagent. This is prepared by dissolving thymol in a little acetic acid and diluting to a convenient strength with concentrated sulphuric acid. The use of the acetic acid avoids the development of the yellow colour which is produced when thymol is dissolved directly in sulphuric acid. The reagent should not be exposed to direct sunlight. The ratio of thymol to titanium may vary within wide limits, but it is found best to have it not less than 100 : 1. If it is necessary to dilute the test solution or standard, concentrated sulphuric acid should be used for the purpose, as the colour fades appreciably when the concentration of sulphuric acid falls below 80 per cent. Fluorides bleach the colour. G. C. J.

Volumetric Determination of Titanium. P. W. Shimer and E. B. Shimer. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 445-451.)—Reduction of titanic to titanous oxide by means of ordinary zinc is very slow. By the use of a reductor described in this paper, the reduction may be accomplished in fifteen minutes. The reductor consists of a tube 3 feet long and $\frac{5}{8}$ inch internal diameter, drawn out at its lower end, which is plugged with glass-wool. Above the glass-wool the tube is packed with amalgamated zinc of a size that passes through a 10-mesh sieve and over 20-mesh. The drawn-out end of the reductor passes through one hole of the double-bored rubber stopper of a flask, and reaches half-way to the bottom of the flask. To the upper end of the reductor a 4-inch funnel is attached. Before commencing a reduction, the flask is filled with carbon dioxide and connected to the reductor, through which a little hot dilute sulphuric acid has been passed to warm the zinc. The solution to be reduced is now placed in the funnel and drawn through the reductor slowly by applying moderate suction to the tube which pierces the second hole in the rubber stopper of the flask. The rate of flow should be such that the solution drops from the reductor and does not form a continuous stream. The reductor is washed by sucking through it hot water acidulated with sulphuric acid, and the flask is finally disconnected; any air contained in it is expelled by passing carbon dioxide through it, and the solution is titrated with ferric solution, using thiocyanate as indicator.

In presence of a large amount of iron, as in a solution of a steel, it is necessary to put the solution twice through the reductor. In pig-iron, the whole of the titanium is found in the residue insoluble in hydrochloric acid. Vanadium, which interferes with the titration of titanium, is most conveniently separated from the latter by fusion with sodium carbonate and sulphur. The aqueous extract of the melt contains all the vanadium, but no titanium. The undissolved residue is then got into solution by appropriate means, and the titanium determined as described.

G. C. J.

Fusion of Certain Rare Earths with Alkali Carbonates. Separation of Tungsten from Iron, Beryllium, and Aluminium. M. Wunder and A. Schapiro. (*Ann. Chim. anal.*, 1912, **17**, 323-327.)—Fusion of oxides of cerium and lanthanum with sodium or potassium carbonates left insoluble residues in close agreement with theory, provided that solid alkali carbonate was added at the time of boiling the mass with water. In the case of thorium oxide, however, the results were somewhat too low, whilst with didymium and erbium oxides a large proportion remained in solution. For example, on fusing 0·1940 grm. of didymium oxide for an hour with 4 grms. of sodium carbonate, boiling the mass for twenty minutes with 200 c.c. of water, to which 1 grm. of sodium carbonate had been added, and filtering the hot liquid, the insoluble residue weighed 0·1629 grm., whilst a crystalline deposit (0·028 grm.) separated as the filtrate cooled (loss = 14·66 per cent.). The insoluble residue when fused again and treated as before yielded 0·1376 grm. of insoluble residue and 0·0251 grm. of deposit in the filtrate (loss = 15·4 per cent.); whilst after a third fusion the loss was 11 per cent. In corresponding tests with potassium carbonate the respective losses of didymium oxide after the three fusions were 8·12, 10, and 11·97 per cent. Erbium oxide fused for an hour with sodium carbonate, and the mass boiled with water and alkali carbonate, showed a loss of 5·4 per cent.; whilst the losses after two successive fusions with potassium carbonate were 36 and 37·5 per cent. respectively.

Separation of Tungsten.—On fusing tungstic oxide for fifteen minutes with about six times its weight of sodium carbonate or potassium carbonate, the mass was completely soluble in water. For the separation of a mixture of tungstic and iron oxides the mixture (about 0·5 grm.) was fused for an hour with about 5 grms. of sodium carbonate, the mass taken up with water, and the liquid boiled for twenty minutes after the addition of 1 grm. of solid sodium carbonate. The undissolved iron was fused again in the acid, and was finally dissolved in hydrochloric acid and precipitated with ammonia. The tungsten in solution was precipitated by mercurous nitrate dissolved in very dilute nitric acid and neutralised by ammonia.

	Taken. Grm.		Found. Grm.
Tungstic oxide ...	0·2983	first filtrate ...	0·2950
		second filtrate ...	0·0023
Iron oxide ...	0·1106	first fusion ...	0·1132
		second fusion ...	0·1102

Good results were also obtained in a single fusion continued for two hours. Tungsten and beryllium oxide were separated in exactly the same manner and with similar good results. In the case of aluminium oxide the mixture, after fusion with sodium carbonate, was treated with ammonium nitrate in excess to separate the two oxides.

C. A. M.

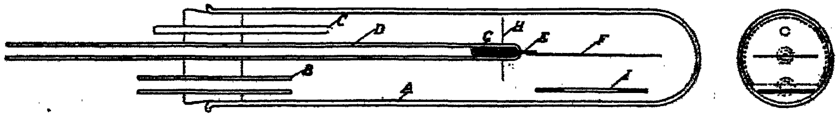
APPARATUS, ETC.

New Calorimeter Bomb, with Special Advantages as to Material of Construction and Method of Operation. S. W. Parr. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 389-393.)—The bomb is constructed from an alloy of nickel, copper, tungsten, and chromium, the composition and general properties of which are described in a paper communicated to another section (sec. 2). The melting-point of the alloy is so high (1300° C.), and the shrinkage at the point of solidification so great, that sound castings are obtained with difficulty, but when obtained, they form an excellent substitute for platinum. Another author (Jesse, vol. i., 233-236) has shown that, in determining the heat of combustion of a sample of coal, not more than 0·2 mgrm. of copper and 0·8 mgrm. of nickel are dissolved by the combined nitric and sulphuric acids present in the products of combustion of coal. The heat of formation of the equivalent quantities of copper and nickel nitrate introduce an error of less than 0·02 per cent. into a calorimetric determination. The other improvement described consists in the use of a rubber gasket in place of the usual lead gasket, and the replacement of the ordinary steel needle valve by a seated valve bearing a rubber gasket. The portion of the gasket which comes in contact with the gases in the bomb is a ring about 0·001 inch in width, and to reach this the hot gases have to pass through a very narrow space, with massive metallic cooling surfaces on each side. There is only indirect proof that the use of rubber gaskets is without effect on the accuracy of the results obtained by means of the bomb. In two series of experiments with sugar and benzoic acid, the greatest deviation from the mean was 7 parts in 10,000; and the ratio of the heat of combustion of benzoic acid to that of sugar was found to be 1·6014, against 1·6003 found by Fischer and Wrede, and 1·6016 the value found by the Bureau of Standards.

G. C. J.

Electrically-Heated Microscope Slide. F. G. Cottrell. (*J. Amer. Chem. Soc.*, 1912, 34, 1328-1332.)—The apparatus has been found useful for the study of liquid crystals and for any observations under the microscope where an adjustment of temperature is desired. The slides are made by cutting, crosswise, ½-inch widths from very thin (0·6 to 0·8 mm.) 1×3 inch microscope slides, the cut edges being rubbed smooth on fine emery cloth or carborundum powder. Both ends of these little slides are then coated with gold or platinum, and the centre part is specially coated with a very thin transparent film of platinum, the platinised ends serving as terminals for the centre of the slide, and the film on the central portion acting as the electrical resistance for heating the slide.

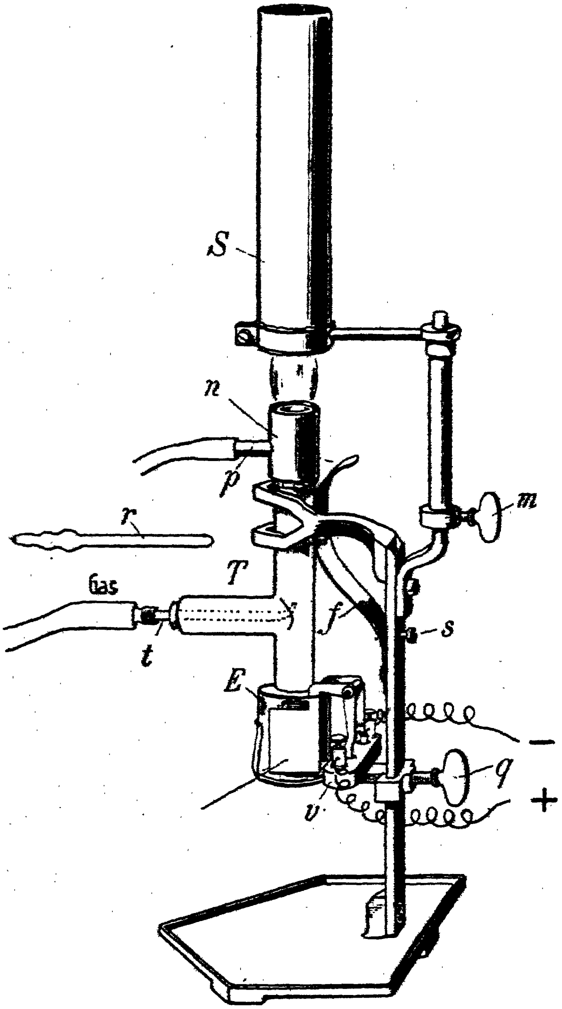
The ends of the slide are painted for about ¼ inch with gold porcelain paint, or dipped in a collodion solution to which a small quantity of gold or platinum chloride has been added, and the adhering film dried in the air. The organic matter is next gradually burned off by means of a Bunsen flame, a brilliant metallic surface being left, which may be thickened if necessary by repeating the process. To obtain the far thinner film required for the centre of the slide, a stout test-tube, *A* (fig.), carrying a rubber stopper, is fitted with a tube, *B*, for connecting to the vacuum pump, an aluminium wire anode, *C*, and a glass tube, *D*, through which is sealed a stout platinum wire, *E*, carrying a welded stiff platinum plate, *F* (cathode). Electrical connection to the last-named is made by running mercury down the glass tube to *G*. The negative wire from the induction coil may be placed in this mercury. A mica plate, *H*, is slipped over the cathode tube to screen the end from deposit and enable the process to be observed as the platinising of the slide *I* proceeds. When the slide has been placed in position, the joints are sealed with melted beeswax containing 10 to 20 per cent. gutta-percha. The vessel is exhausted to 0·01 mm. of mercury, and this point is indicated by the green fluorescence of the glass when the current is applied. As soon as a slight but perceptible darkening of the central portion of the glass slide is observed, it is withdrawn. An ordinary glass slide (3 inches × 1 inch), fitted with binding posts and metal springs, forms a convenient holder for these platinised slides. The rheostat used with these slides consists of a glass cylinder (1 inch × 4 inches high) on a substantial foot. Through a cork stopper pass two glass-stemmed platinum electrodes similar to the cathode in the figure, except that the platinum sheet may be omitted. One of the electrodes should reach to the bottom of the cylinder, the other sliding easily through the cork to allow adjustment of the distance between them. The cylinder is filled with distilled water, acidified with 1 drop of dilute sulphuric acid. For temperatures only slightly above room-temperature, only very faint acidity may be needed. In observing electrolytes, a thin cover-glass may be placed over the platinum surface, but in other cases the material is in actual contact with the slide. The temperature is easily controlled. Probably the temperature could be ascertained by measuring the current flowing through the platinum with a galvanometer, while measuring drop in potential across the film by the compensation method, and from these data



One-fifth natural size.

estimating the resistance of the film, which, in the case of pure platinum, should be directly proportional to the absolute temperature. A. R. T.

Sodium Flame Lamps for Polarisation. E. Beckmann. (*Ber.*, 1912, 45, 2523-2529.)—The objections to the ordinary form of polarisation lamp, in which solid sodium chloride is volatilised in a Bunsen flame, have led the author to design lamps in which the sodium coloration is obtained by the minute globules of liquid projected from the surface of a solution of sodium hydroxide or carbonate which is undergoing electrolysis. Such a lamp is illustrated in the figure. The body of the burner is constructed of porcelain or quartz-glass, and the gas is introduced through a removable side-tube having a small hole, *r*, which serves as a jet. By turning the jet more or less from the vertical direction the amount of air sucked in at the bottom may be regulated. The electrolyte contained in the vessel situated below the burner consists of a 2 to 4 per cent. solution of sodium hydroxide or carbonate, and plate electrodes of nickel are employed. Two accumulators in series or three dry cells provide the current, and the evolution of gas must be so controlled that the liquid appears merely milky, without the formation of large bubbles. If the electrolyte vessel be brought sufficiently close to the bottom of the burner-tube, the whole of the gas evolved is drawn in, and the intensity of the light is equal to that of the ordinary salt burner. The intensity of the flame may be increased by fitting a porcelain or iron shell around the top of the burner, as shown at *n*, forming an annular orifice at the top, and closed at the bottom. A stream of oxygen, not under any appreciable pressure, is conducted through this annular shell. Another form of burner is also



described, in which the electrolytic bath surrounds the top of the burner, and only the outer zone of the flame is coloured by the sodium solution. When a flame of unusual intensity is required, a burner suitably modified to burn hydrogen instead of coal-gas is employed. J. F. B.

Determination of Molecular Weights of Volatile Liquids. C. W. Porter. (*J. Amer. Chem. Soc.*, 1912, **34**, 1290-1293.)—In the Victor Meyer, Gay-Lussac, and Hofmann methods for determining molecular weights, a weighed quantity of the substance is volatilised at a fixed temperature and pressure, and the volume of the vapour is determined by measurement. In other methods the volume and temperature are fixed, and the pressure is determined, or the weight of a known volume of the vapour is estimated, as in Dumas' method. The remaining possibility—namely, the estimation of the molecular weight of a substance by noting the temperature when the volume, pressure, and weight are known and fixed—has been utilised by the author as the basis of his method. A glass bulb of about 300 c.c. capacity is immersed in a glycerol and water-bath contained in a glass jar tubulated near the bottom. A tube of small bore is sealed on each end of the bulb, and one of these passes through a stopper in the tubulure of the jar, and is connected by rubber tubing to a mercury reservoir. The tube at the upper end of the bulb is connected by rubber tubing with a short capillary tube, and this latter is sealed by a solid glass plug and rubber connection. The glycerol bath is electrically heated by means of coils of "nichrome" wire. The volume of the bulb from the plug at the top to a mark on the tube at the lower end is accurately determined. Mercury is poured into the reservoir until the level in the tube is at the mark just mentioned, when the bulb is completely filled with mercury by pressure applied at the top of the mercury reservoir with an air-pump. Before the mercury reaches the top of the tube, a small sealed glass bulb containing a weighed quantity of the liquid to be examined is dropped on to the mercury, so that the long capillary end of the bulb will be directed upwards. The capillary tube fitting on to the top tube of the large glass bulb is then placed in position, held by heavy rubber tube, and wired on. Pressure is then applied till mercury appears at the top of the capillary tube, when the solid glass plug is inserted and the rubber connection wired on. The mercury will have forced the end of the bulb holding the test-liquid into the capillary bore of the attached tube, and, by bending this slightly at the connection, the end of the small bulb may be broken off, when the vapour from the liquid will be free to escape. The bath is next heated and vigorously stirred. When the mercury has been forced down to the mark on the lower tube, the vapour in the large bulb is at atmospheric pressure, and a thermometer in the bath registers the temperature. To obtain exact measurements, the heating is continued till the level in the tube is a little lower than the mark; then the apparatus is allowed to cool slowly and the temperature read off when the level is exactly at this mark. When the rising and falling column reaches this mark at a temperature constant to 0·5° C., the reading is recorded. The volume of the bulb must be corrected for the temperature attained, and allowance made for vapour tension by deduction from the barometric pressure. The results obtained compare favourably with those yielded by the older methods. A. R. T.

Modified Soxhlet Extraction Apparatus with Distillation Arrangement. F. Friedrichs. (*Zeitsch. angew. Chem.*, 1912, 25, 2208.)—The condenser is arranged eccentrically above the extraction vessel, and can be shut off from it by rotation of the hollow ground-in stopper which closes the extraction vessel. This stopper is constricted underneath, so that it delivers the liquid in the axis of the cartridge. When extraction is complete, the stopper is rotated, and the flow of liquid from the condenser diverted, through a stopcock opened for this purpose, to a side flask. The extract is thus obtained freed from solvent. The whole apparatus may be fixed, as it is not necessary to detach the condenser in order to introduce or remove the substance. O. E. M.

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REPORTS.

Report to the Local Government Board on the Nature of the Colouring Matter of Flour and its Relation to Processes of Natural and Artificial Bleaching. G. W. Monier-Williams. (*Food Reports*, No. 19, 1912.)—The present work is a continuation of that described by Hamill and the author in a previous report on the bleaching of flour (ANALYST, 1911, **36**, 254). The colouring matter of flour is either carrotene ($C_{40}H_{56}$) or a substance, or mixture of substances, so closely allied to it that the absorption spectra are practically identical. The colour of carrotene can be discharged either by atmospheric oxygen or by nitrogen peroxide, but these two processes are distinct, and result in the formation of different substances. Pure carrotene, on exposure to air, is bleached by absorption of oxygen, no oxides of nitrogen being absorbed from the air. It is only reasonable to suppose that the natural ageing of flour is a similar process, and that in the bleaching of flour by nitrogen peroxide certain substances are produced which are dissimilar to those formed during the natural ageing of flour. Although these substances are present in exceedingly minute amounts, it would appear that artificially bleached flour is not quite the same as flour that has been naturally aged.

The amount of nitrite-reacting substance present in flour tends to increase on storage, although not to any great extent. Even in unbleached flours which had been exposed to the impure air of Widnes, highly contaminated with acid fumes, the maximum amount found was 1·4 parts as sodium nitrite per million. Whether the slight increase in nitrite-reacting substance is due wholly to absorption of oxides of nitrogen or nitrites from the air, or to changes occurring in the flour itself, is not clear. It is noteworthy that unbleached flours showed on the average an increase of nitrite from 0·4 to 1·2 parts sodium nitrite per million, while the bleached samples, which already contained 1·6 parts per million, showed no increase at all except in one instance. The results given by the Griess-Ilosvay test must therefore be interpreted with caution, and from the experiments recorded it appears extremely improbable that unbleached flour stored under ordinary conditions will show more than 1·5 to 2·0 parts per million of sodium nitrite by this test. The report concludes with photographic reproductions of the absorption spectra of the petroleum ether extracts of a bleached and unbleached flour, and of a solution of carrotene in the same solvent. Short addenda are attached—(1) on the effect of prolonged storage upon the nitrite content of highly bleached flour, and (2) on the effect of excessive bleaching upon the baking qualities of flour. H. F. E. H.

Metropolitan Water Board. Sixth Annual Report. A. C. Houston.—This Report contains the results of the chemical and bacteriological examination of the London waters for the twelve months ended March 31, 1912. A considerable amount of the water abstracted from the Rivers Thames and Lea is now stored before filtration, and numerous results are given, of which the following may be cited, illustrating the effect of storage, and also the effect of the subsequent sand filtration of the stored water.

Parts per 100,000.	Thames, before Storage.	Thames, after Storage.	Thames, after Filtration.
Ammoniacal nitrogen	0·0073	0·0024	0·0002
Albuminoid "	0·0155	0·0109	0·0059
Oxidised "	0·26	0·25	0·25
Chlorine	1·76	1·71	1·70
Oxygen absorption, three hours at 80° F.	0·2062	0·1458	0·0892
Turbidity	3·33	0·81	Nil
Colour, mm. brown in 2-foot tube ...	69	39	19
Total hardness	21·67	21·33	21·00
Permanent hardness	5·62	5·40	6·09
Bacteria per c.c.: gelatine	9,155	1,079	10·9
" " agar	356	47	1·7
" " bile-salt agar ...	48	7	—

The author strongly emphasises the value of storage as improving the quality and increasing the safety of the water-supply. The test for the presence of spores of the *B. enteritidis sporogenes* is not now applied to the filtered waters, as they practically always yield negative results. In order to obtain a rapid provisional idea of the quality of the water sent into consumption each day, the five minutes' oxygen absorption test at 80° F. is made, and a result of 0·038 per 100,000 (experimentally arrived at) is taken as the standard which a sample should not exceed.

The author concludes with a review of the position of the London water-supply, in which he states that the river waters are undoubtedly unsatisfactory in quality, and the judicious selection of water is becoming increasingly difficult, as the consumption increases while the volume of the rivers remains constant. The levelling effect of storage, however, renders the possibility of any sudden dangerous pollution a matter of less grave concern than would otherwise be the case. But it is unwise to abstract water from the rivers independently of its quality, and therefore the adoption of certain supplementary processes of purification antecedent to filtration should be considered in the future. J. H. J.

REVIEWS.

THE FREEZING-POINT, BOILING-POINT, AND CONDUCTIVITY METHODS. Second edition. By HARRY C. JONES. Easton, Pa.: The Chemical Publishing Company. 1912. Price 4s.

The name of the author is a sufficient guarantee of the high character of this work, and the fact that this is the second edition shows that its publication has supplied a real want. The author deals with the theory of the freezing-point and boiling-point methods, and with their practical application to the determination of molecular weights, and also explains fully how these methods, as well as the conductivity method, may be applied to the measurement of electrolytic dissociation. Both the theoretical and practical sections are clearly written, and the experimental procedure is described in such a manner that, with the help of the well-chosen illustrations, no one accustomed to the use of ordinary physical apparatus should have any difficulty in making the determinations. Year by year increasing demands are being made on the technical chemist, and operations which were formerly carried out only in the laboratories of colleges and other teaching institutions have now become matters of almost everyday procedure in many technical laboratories. In such laboratories, therefore, this little book may be found very helpful, and chemists generally will find in it, in a concentrated form, information for which they would otherwise have to make somewhat extensive search. A. CHASTON CHAPMAN.

ALLEN'S COMMERCIAL ORGANIC ANALYSIS. By DAVIS and SADLER. Vol. VI. London: J. and A. Churchill. 1912. Price 21s. net.

Corresponding roughly with Vol. III., Part 2, of the last edition, this volume deals with amines, tar bases, the vegetable alkaloids and their preparations, and the plants from which they are derived. So much work has been done on the chemistry of these substances since 1892 that much of the book has had to be entirely rewritten, and the task had been advisedly, if not of necessity, placed in the hands of a number of workers who may be regarded as specialists in their particular subjects.

The general plan of the old volume has been closely adhered to, but the uniformity of style and treatment inherent in a *one man* book has, to a certain extent, been lost. In spite of this, the changes in some of the sections—*e.g.*, that devoted to the general reactions of the alkaloids—are surprisingly small, and paragraph after paragraph appears almost unaltered. While the sections dealing with the toxicology of individual alkaloids have in most cases been rewritten or substantially added to, the treatment is hardly as systematic as could be desired. In some cases, for instance, full information as to medicinal and poisonous doses is given, while, in others, it is entirely omitted. Although the work does not pretend to be a treatise on toxicology, the general processes for the separation, detection, and estimation of the highly toxic alkaloids when present in organic mixtures, might well have been treated at greater length once for all, instead of repeating the description of the preliminary treatment of organic remains at least four times in connection with different alkaloids.

As regards the majority of the alkaloids, recent progress has obviously been made, mainly on the purely scientific side, and the chemical constitution and relations of these bodies has received more attention than the methods for their detection and estimation.

The commoner alkaloids, their salts, the crude drugs from which they are obtained, and the various pharmacopœial preparations containing them are very fully dealt with, and the numerous methods which have been put forward in the British and other pharmacopœias for the assay of drugs, such as opium, are described in ample detail, and some attempt is made to aid the reader in deciding among the competing processes. The sections on tea, coffee, and cocoa have, on the whole, been brought well up to date, and are eminently practical. They contain, however, a somewhat large proportion of obsolete matter, and it comes as a shock to our ideas of the progress of analytical chemistry to find that three-quarters of the analytical tables and of the methods of estimation still in use are, at least, ten years old, and four-fifths date from more than twenty years ago.

In connection with cocoa the numerous methods for the determination of shell receive scant attention, and the paper of Ulrich on this subject, and also Ewer's method for the determination of starch, might well have received mention.

An innovation on the last edition is the introduction of illustrations of the microscopical structure of tea and coffee. In a work of this importance such illustrations may well have a place, but it is not unreasonable to expect that they should be prepared specially for the book, and they should certainly not be confined to two of the best-known substances, neglecting all those with which we are less familiar, and in connection with which some plates would have been really valuable.

The work as a whole suffers somewhat, as is perhaps inevitable, from the system on which it is written, the authors of some of the sections apparently not being aware of what has appeared in others, so that occasionally contradictory statements occur, as, for instance, theobromine is said to sublime at 220° C. in the article on cocoa, and at 290° C. in the chapter on the alkaloid itself.

Apart from these small defects, the volume as a whole presents a most useful and up-to-date résumé of the subjects dealt with, the type is excellent, printers' errors are few, and the text clear and concise. CECIL H. CRIBB.

VINS. Par U. GAYON et J. LABORDE. Paris: Ch. Beranger. 418 pp. + Table of Contents, etc. 1912. Price 7 f. 50.

This volume forms one of a series of "Manuels Pratiques D'Analyses Chimiques," which is intended mainly for the guidance of official (French) analysts and experts, and is issued under the joint editorship of MM. Bordas and Roux. It would be difficult to find two experts who could write with greater experience and knowledge of, and authority on, French wines in general and Gironde wines in particular. One naturally expects from men who combine a rare knowledge of wine chemistry with the art of the wine "connoisseur" something more than a mere statement of analytical methods and results, and in this regard the work under review is no disappointment. From their remarks on the wider objects of wine analysis and on wine from a legal point of view, it is evident that the authors have little sympathy with the narrow analytical standpoint, which is regardless of the genuine difficulties and requirements of a great industry. While it is clear that certain standards are necessary, it is made equally obvious that such standards should be applied without prejudice and partiality, and only as the result of a very wide practical experience. The authors draw attention to an interesting distinction between the legal definition of wine and the commercial definition. Substantially the French legal definition of wine is that it shall be derived exclusively from the fermentation of fresh grapes or of fresh grape-juice. The commercial definition of a satisfactory wine is that it shall be *loyal et marchand*. For a wine to be *loyal et marchand* it must be a natural product, sufficiently well constituted, without hidden defects, and its organoleptic properties must be beyond reproach. Obviously this definition is more satisfactory than the legal one. Every wine that conforms to the commercial definition passes the legal standard, but there may be many legal wines which are by no means satisfactory from the commercial, or, for the matter of that, from the consumer's point of view. In the first part of the work, and under the general heading "Wine from a Legal Point of View," the authors give a résumé of wine legislation in the most important countries, and naturally enough, the information regarding French legislation is very complete. Of particular interest to analysts will be the information regarding the limitations imposed by the French law on the addition of substances normally present in wine and their use in wines deficient in regard to the same, for this is a debatable ground, and the French legislature has, while maintaining its general definition, not excluded the possibility of "ameliorating" by reasonable means a product which would otherwise be commercially worthless. In this connection one may instance the permission to use, within certain limits, tartaric acids in musts which are lacking in acidity. Such musts, if fermented without any addition, almost invariably become the prey of undesirable bacterial activity. In the second chapter the French official methods of analysis are dealt with, and in the third chapter a complete scheme for analysis is set forth. Criticism of analytical methods which have become standardised is liable to be misleading, but so far as such criticism is permissible, the reviewer is of opinion that some of the French methods—for instance those for estimating total acidity, sugar and glycerol respectively—are scarcely as satisfactory as the official German methods. In the succeeding chapter the more ordinary methods of adulteration are described, and methods for determining such adulteration dealt with. Following a chapter dealing with special wines and wine diseases we have an appendix, the first part of which sets out fully in chronological order the laws and decrees constituting French wine legislation. The second half comprises a number of tables giving the results of analyses of French wines from various districts, together with a number of tables of a more special analytical character. The work contains four sheets of microphotographs showing various wine diseases, and seven illustrations in the text.

P. SCHIDROWITZ.

L'ANALYSE DES VINS PAR VOLUMÉTRIE PHYSICO-CHIMIQUE. Par PAUL DUTOIT et MARCEL DUBOUX. Lausanne: F. Rouge and Co. 186 pp. + Table of Contents + 43 illustrations in text. 1912. Price 5 f.

The title of this work scarcely does justice to its contents, for the latter constitute a record of a large number of apparently most carefully conducted experiments on the application of physical methods to the elucidation of analytical problems. Briefly put, Professor Dutoit and his colleague appear to prove most satisfactorily that the conductivity method can be employed for many and diverse quantitative determinations. Much of the work here recorded appears to be unpublished, but as the bulk of it has appeared in journals which are not readily accessible to the analyst in this country, it is probable that the contents of the book as a whole will come as something of a surprise to those who have not been in a position to devote special attention to this subject. The principle employed by the authors is that of combining ordinary volumetric methods with conductivity measurements, or, put very briefly, a solution of the reagent to be employed is run in by means of a burette, and the alteration in conductivity of the solution containing the constituent to be determined is observed. In this manner curves are obtained which show distinctly the saturation points. It is scarcely practicable within the limits of a review to describe or criticise adequately the methods or results obtained by the authors, but it may be sufficient to state that the mass of details set forth by the authors indicate clearly that determinations such as those of chlorides, sulphates, phosphates, lime, total tartaric acid, total acidity, total alkalinity, ash, ammonia, and so on, may be carried out satisfactorily and rapidly. The control figures obtained by gravimetric methods, which are given by the authors, appear to prove beyond doubt that the principle of the method is a reliable one. For those who have to carry out a number of special determinations under routine conditions, the method appears to be an ideal one, and the work can be confidently recommended to every analyst and to those engaged in analytical operations as being not only out of the common, but as one of which the study may result in the saving of much time. P. Schidrowitz.

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NEW BOOKS.

Portland Cement: Its Composition, Raw Materials, Manufacture, Testing, and Analysis. By Richard K. Meade. Second edition. Chemical Publishing Company, Easton, Pa., U.S.A. 1911. London: Williams and Norgate. Price $4.50.

Octavo volume, containing 501 pages of subject-matter, with 169 illustrations and numerous tables, followed by an index. The subject-matter is classified as follows: I. Relation between Mortar Materials, and History of the Development of the American Portland Cement Industry. II. Nature and Composition of Portland Cement. III. Raw Materials. IV. Proportioning the Raw Materials. V. Quarrying, Excavating, Drying, and Mixing the Raw Materials. VI. Grinding and Grinding Machinery. VII. Burning: Kilns and Processes. VIII. Fuel, and Preparation of the Same. IX. Cooling and Grinding the Clinker; Storing and Packing the Cement, etc. X. Analysis of Cement. XI. and XII. Analysis of Cement Mixtures and Raw Materials. XIII. Inspection of Cement. XIV. Specific Gravity. XV. Fineness. XVI. Time of Setting. XVII. Tensile Strength. XVIII. Soundness. XIX. Detection of Adulteration in Portland Cement. XX. Investigation of Materials for Manufacturing Portland Cement.

FERTILITY AND FERTILISER HINTS. By J. EDWARD HALLIGAN. Chemical Publishing Company, Easton, Pa., U.S.A. London: Williams and Norgate. Price $1.25.

Octavo volume, containing 143 pages of subject-matter, with 12 illustrations and an index. The subject-matter comprises: I. Chemical Elements needed by Plants, and the Composition of Plants. II. Fertility of the Soil. III. Maintaining Soil Fertility. IV. Farm Manures. V. High-Grade Nitrogenous Materials. VI. Low-Grade Nitrogenous Materials and Functions of Nitrogen. VII. Phosphates. VIII. Superphosphates: Effect of Phosphoric Acid. IX. Potash Fertilisers. X. Miscellaneous Fertiliser Materials. XI. Lime, Gypsum, and Green Manures. XII. Commercial Fertilisers. XIII. Valuation of Fertilisers. XIV. Home Mixtures.

A BRIEF LABORATORY GUIDE FOR QUALITATIVE ANALYSIS. By ARTHUR E. HILL, Ph.D. Chemical Publishing Company, Easton, Pa., U.S.A. 1911. Price $1.

Small octavo volume, containing 76 pages of matter and an index.

SMOKE: A STUDY OF TOWN AIR. By JULIUS B. COHEN, Ph.D., F.R.S., Professor of Organic Chemistry in the University of Leeds; and A. R. RUSTON, B.A., B.Sc. London: Edward Arnold. 1912. Price 5s. net.

Octavo volume, containing 86 pages of subject-matter, with 35 illustrations and an index. The subject-matter is arranged as follows: I. Solid Products of Combustion. II. Gaseous Impurities. III. Town Fog. IV. Dispersal of Soot. V. Influence of Coal Smoke upon Health. VI. Analysis and Manuring Value of Soot. VII. Analyses of Leeds Rainwater. VIII. The Sootfall of London.

LECTURES DELIVERED AT THE CENTENARY CELEBRATION OF THE FIRST COMMERCIAL GAS COMPANY TO SELL GAS AS AN ILLUMINANT. Held at the Franklin Institute, Philadelphia, Pa., April 18 and 19, 1912. Edited and published by the American Gas Institute, New York City.

Octavo volume, containing 174 pages of subject-matter, with 43 illustrations.

THE EXTRA PHARMACOPŒIA OF MARTINDALE AND WESTCOTT. Revised by W. HARRISON MARTINDALE and W. WYNN WESTCOTT. Fifteenth edition. In two volumes. London: H. K. Lewis. 1912. Price, Vol. I., 14s. net; Vol. II., 7s. net.

This work has increased still further in size since the fourteenth edition appeared, and now fills two volumes. Vol. I. contains 1,114 pages of matter, inclusive of the alphabetical index of subjects and posological table. There is also a Schedule of Poisons, and a Therapeutic Index of Diseases and Symptoms, and this is followed by a list of poisons and their antidotes. Vol. II. contains 370 pages of matter, with: I. Analytical Addenda to Materia Medica: Official and Non-Official. II. Synthetic Notes: Physiological Effect in Comparison with Chemical Constitution. III. Physiological Standardisation, etc. IV. Mineral Water Table, with Proportions of Active Ingredients. V. Notes on Antiseptic Power of Chemicals. VI. Analytical Memoranda. VII. Water, Milk, and Butter Analysis. VIII. Carbon Monoxide and Dioxide Tests. IX. Ptomaines. X. Stomach Contents, Examination, etc. XI. Bacteriological Water Examination. XII. Action of Acids on Common Metals and their Oxides: A New Table. XIII. Glossaries of Words and Phrases in Foreign Prescriptions in the Danish, Dutch, French, German, Italian, Portuguese, and Spanish Languages; Tables of Weights and Measures of Approximate Equivalent Doses, and of the Metric Weights and Measures and their Equivalents in the British Pharmacopœia.